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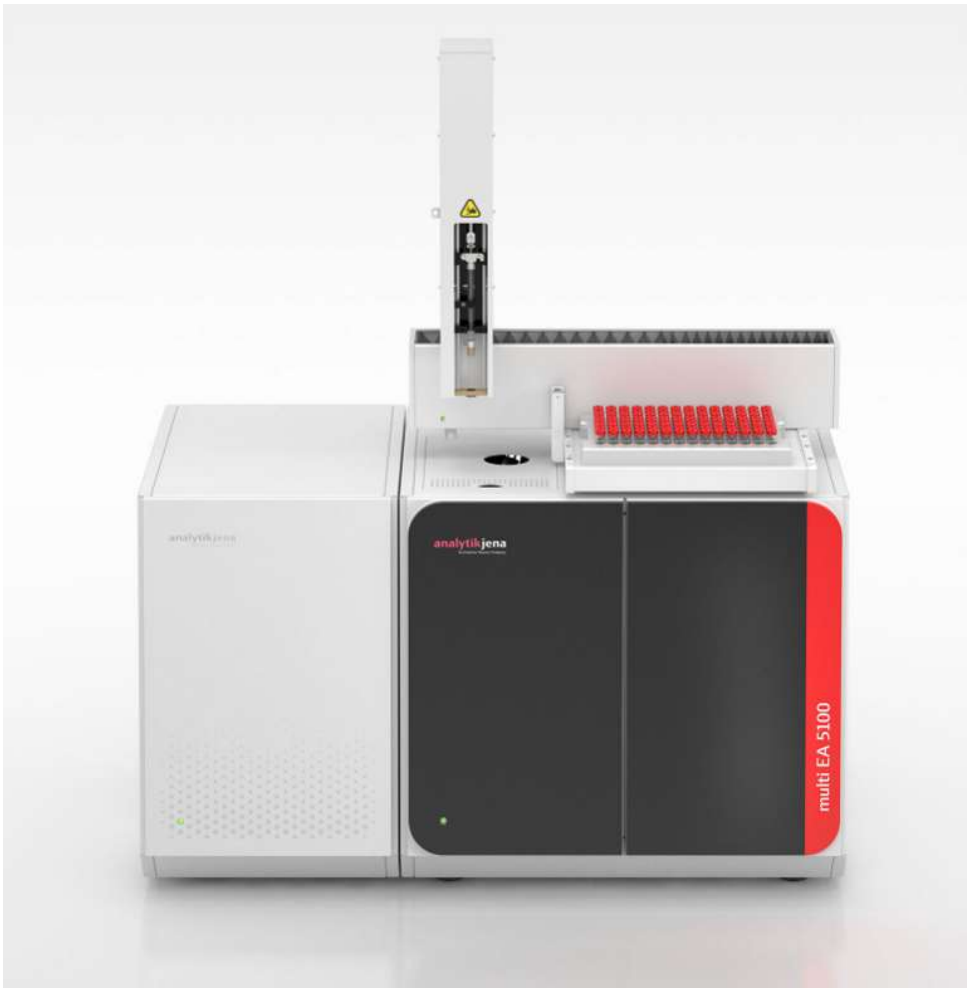
Operating Manual

multi EA 5100
C/N/S/X Analyzer



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

General Information

http://www.analytik-jena.com

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Technical Documentation

Analytik Jena GmbH+Co. KG

Table of contents

1 About this user manual

The multi EA 5100 analyzer is intended for operation by qualified personnel with the aid of this user manual.

The user manual contains descriptions of the design and operation of the multi EA 5100 and provides personnel experienced in analysis with the necessary information for the safe handling of the basic module and its components. The user manual also includes notes on the maintenance and care of the equipment and potential causes and remedies for any faults.

Conventions

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

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

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

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

Symbols and signal words used in this manual

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

WARNING

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

CAUTION

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

NOTICE

Provides information on potential material or environmental damage.

2 Intended use

The multi EA 5100 is a modular analyzer for determining the sulfur, nitrogen, chlorine and carbon content in solid, liquid, paste-like, viscous and gaseous samples. Sample digestion is achieved via pyrolysis and subsequent thermal oxidation of the achieved result of the pyrolysis. The various element contents are determined in accordance with national and international standards by subsequent selective detection systems.

Depending on the functionality, the basic module includes at least one detection module and one sampling module. The basic module and any connected modules are controlled by the multiWin control and analysis software, and the analysis of the measurement data is performed in this as well.

The analyzer 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.

Suitable sample types for the multi EA 5100

The analyzer can be used for the following sample types:

- Organic solids, e.g., wax, polymer
- Organic liquids, e.g., fuels, solvents
- High-viscosity organic liquids, gels and paste-like samples, e.g., crude oil, asphalt, bitumen, tar
- Organic gases and gas mixtures, e.g., natural gas, methane
- Compressed liquefied gases, e.g., LPG, NPG, butane, propane, propylene
- Carbon modifications, e.g., coal, elemental carbon, soot, coke
- TC/TOC/TIC/NPOC in water analyses, e.g., waste water
- EC/OC determination in particulate emissions, e.g., air quality control, three-way catalytic converter research
- AOX and EOX for water, sludge and soil via the column or batch method

Unsuitable samples

The following sample types may not be analyzed with the multi EA 5100:

- Highly flammable organic compounds – risk of explosion!
- Self-reactive materials and explosives, e.g., peroxides, epoxides, azides
- Organic and inorganic phosphorous compounds, e.g., phosphoric acid ester
- Silico-organic and reactive or unstable inorganic silicium compounds, e.g., silane
- Metal organic compounds, e.g., nickel carbonyl
- Corrosive or highly-reactive substances, inorganic acids, HF, Cl_2, H_2SO_4
- Materials with high inorganic or organic salt content, in particular Na^+ und K^+ ions, e.g., soil, fertilizers, feed, construction material
- Inorganic compounds, e.g., minerals, inorganic halogens
- Samples requiring more than 1100 °C for complete oxidation or digestion, e.g., metals, alloys, N_2
- Pure elements, e.g., sulfur, nitrogen
- Sample with element contents exceeding the permissible operating range.
- TOC, TIC or TC analyses of solids, e.g., soil, sediment

Alkali salts and alkaline earth salts lead to crystallization of all quartz glass components This process is accelerated by increased salt concentrations and combustion temperatures, i.e. reducing the service life of the quartz glass components.

Gases used

The device must only be used with argon and oxygen of the required quality as a carrier gas.

Sum parameters

Attaching the corresponding detectors and sampling modules allows the multi EA 5100 analyzer to determine the following parameters as sum parameters.

Parameter	Description
TS	Total sulfur Organically bound sulfur is detected. Inorganic sulfur compounds are only digested partially or not at all.
TN	Total nitrogen Organically bound nitrogen is detected. Inorganic nitrogen compounds and molecular nitrogen (N_2) are only digested partially or not at all.
TC	Total carbon The total organic carbon and elementary carbon and contained CO_2 is detected. Inorganic carbon compounds are only digested partially or not at all.
TX	Total chlorine The organically bound chlorine contained in the sample is detected. Bromide and iodine compounds are only detected partially. Fluorine compounds are not detected at all. The result is output as defined as total chlorine. Inorganic halogen compounds are only digested partially or not at all.
EOX	Extractable organically bound halogens The EOX parameter is the sum of organically bound halogens (chlorine, bromide, iodine) extracted under defined conditions from a water sample or from solids (sludge and sediment) using an organic solvent. Fluorine is not detected.
AOX	Adsorbable organically bound halogens The AOX parameter is the sum of organically bound halogens (chlorine, bromide, iodine) adsorbed under defined conditions (pH < 2 with HNO_3) from a water sample or a solid sample (sludge and sediment) in active carbon using the vibration or column method. Fluorine is not detected.
EC/OC	Elementary carbon/organic carbon The content of elementary carbon of a sample is detected directly after the thermal desorption of the organic carbon. The amount of organic carbon is then determined sequentially after thermal oxidation of the remaining sample.

The following parameters can be determined from aqueous samples with the multi EA 5100:

Parameter	Explanation
TC	Total carbon
TIC	Total inorganic carbon The total inorganic carbon from carbonates, hydrocarbonates and dissolved carbon dioxide is determined.
TOC	Total organic carbon The total organic carbon content is determined mathematically by calculating the difference: TOC = TC - TIC. Determination of the TC and TIC content of the sample is required for this.
NPOC	Non purgeable organic carbon The total non-purgeable organic carbon content of a sample is detected. Highly volatile organic compounds are not or only partially detected.

3 Security

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

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

3.1 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 dangerous electrical voltage	In the device interior on the electronics cover Warning against electrical voltage. Before opening the electronics the device must be disconnected from the mains.
	Warning about hot surface	In the device interior on the combustion furnace On the top cover of the combustion furnace On the heated gas transfer line from the Cl module 5100 Touching the furnaces or the ends of the gas transfer lines during operation or shortly after shutdown of the device can result in burn injuries.
	Warning against corrosive substances	On the "high sensitive" measuring cell of the Cl module 5100 Exercise caution when handling the electrolyte solution. It contains highly-concentrated acetic acid.
		On the sulfuric acid container and the safety attachment of the Cl module 5100 Exercise caution when handling concentrated sulfuric acid
	Warning against UV radiation	In the S module 5100 (basic. MPO) Switch off the detection module before performing maintenance on the UV lamp.
	Caution (GHS07)	On the absorber and the chemical ozone decomposer of the N module 5100 On the chemical ozone decomposer of the S module 5100 MPO

Warning symbol	Meaning	Comment
		The cartridges contain hazardous substances. Do not open the cartridges. Only replace the entire cartridge.

Mandatory action labels	Meaning	Comment
	Disconnect the power supply before opening the device cover.	On the rear panel of and/or the side panels of the basic module and the detector modules In the device interior on the electronics cover
	Observe the operating manual	On the rear panel of and/or the side panels of the basic module and the detector modules

3.2 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.

3.3 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.

3.4 Safety instructions: during operation

3.4.1 General safety instructions during operation

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.
- Free access to the power switch on the back of the enclosure must be ensured 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.
- The furnace operates at temperatures of 700 to 1100 °C. Hot components must not be touched during or directly after the operation of the device.
- Caution when handling glass components. Risk of broken glass and therefore risk of injury!
- Keep all combustible materials away from the device.
- When operating the sampling modules (Autoinjector, MMS and ABD), there is a crushing risk for hands and fingers. Maintain a safe distance.
- When handling the Autoinjector, there is a danger of stabbing yourself in the hand or finger with the syringe. Exercise caution when handling the syringe.
- When operating the multi-matrix autosampler with temperature control function (MMS-T or Multi Matrix Sampler with Liquids kit TMP), the syringe holder and the sample tablet achieve temperatures of up to 80 °C during heating. These hot components present a risk of burn injuries.

3.4.2 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!

3.4.3 Safety instructions: electrical equipment

Lethal voltages may occur in the device! 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!
- 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.
- Before opening the device, the device must be switched off at the main switch and the power plug must be disconnected from the power outlet! The only exception is for instructions that explicitly indicate that the doors of the basic module or of a detection module must be opened during operation. This is the case, for example, during the end point routine of the Cl module 5100 or when searching for a gas leak in the measuring gas connection between the basic module and the Cl module 5100.
- Work on the electronics may only be carried out by the customer service of Analytik Jena and specially authorized technicians.

3.4.4 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.

Hazardous substances are used during operation of the following detection modules:

Detection module	Hazardous substance	Use
Cl module 5100	Concentrated sulfuric acid	Drying agent in the sulfuric acid container
	Acetic acid	Preparing the electrolyte solution
	Concentrated nitric acid	
	Methanol	
	Thymol	
S module 5100 coulometric	Acetic acid	Preparing the electrolyte solution
TOC module 5100	40 % orthophosphoric acid	Reagent in the TIC reactor
	0.2 mol/l hydrochloric acid	Reagent for NPOC determination

Acetic acid vapors causing strong irritation of the airways can occur in the measuring cell of the Cl module 5100. Connect the measuring cell with the exhaust hose and connect the detection module to the laboratory exhaust unit.

3.4.5 Decontamination after soiling

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.
- If a Cl module 5100 is used: Concentrated sulfuric acid is used as a drying agent in the detection module. The electrolyte solution in the "high sensitive" measuring cell has a high concentration of acetic acid. To decontaminate, rinse out the sulfuric acid container with its safety attachment and the measuring cell with distilled water.

3.4.6 Behavior during overpressure faults (0206, gas pressure fault)

Extreme caution is required during system overpressure! Incorrect operation can endanger the operating personnel and damage the analysis system. If an overpressure fault is detected, a warning message will appear in the multiWin program.

Observe the following:
- Never switch off a device subject to overpressure!
- Do not introduce any samples.
- Do not close the program.
- Do not switch off the modules.
- Do not switch off the gas supply.
- **N/S/C branch**: Wait until the overpressure in the system has dropped to normal pressure. The routine for reducing the overpressure in the N/S/C branch runs automatically.
- **Chlorine branch**: For devices with an operational Cl module 5100, ventilation must be performed manually (→ "Recommissioning after emergency shutdown (Cl module 5100)" 🖹 40).
- After relieving the overpressure remove the obstruction in the gas path.

3.5 Behavior during emergencies

Observe the following:
- 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 oxygen gas supply as soon as possible after switching off the equipment!
- When closing the argon gas supply, a return of the acid used can occur in the Cl module 5100. For this reason, the measuring gas lines to this module must be disconnected before closing the argon gas supply.
- When attaching the Cl module 5100: There is a risk of injury from sulfuric acid! During an emergency shutdown the vacuum may draw sulfuric acid into the transfer line and the auto-protection valve assembly during the cooling down pe-

riod. After an emergency shutdown, check that the transfer line and the auto-protection valve assembly are not contaminated with sulfuric acid (→ "Recommissioning after emergency shutdown (Cl module 5100)" 🖹 40) before recommissioning.

- When closing the gas supply, a return of the slightly acidic electrolyte solution and the phosphoric acid in the TIC reactor can occur in the S module 5100 coulometric and the TOC module 5100. Check the measuring gas hoses for contamination before recommissioning. Observe the safety instructions for handling acidic solutions during cleaning.

3.6 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.
- Do not use any organic solvents or abrasives to clean the device. Exercise caution when decontaminating the device with disinfectants containing alcohol. The alcohol can damage the safety labeling on the device.
- All maintenance and repair work on the device must only be carried out when the device is switched off (unless specified otherwise).
- Maintenance work and the replacement of system components (e.g., the removal of the combustion tube) must only be carried out when the device has cooled down sufficiently.
- The gas supply must be shut off before performing any maintenance or repair work (unless specified otherwise).
- All protective equipment must be reinstalled and checked for proper function when the maintenance or repair work is complete.
- Ensure that all connections are gas-tight again after maintenance.
- 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.
- During maintenance of the combustion tube and of the auto-protection valve assembly, there is a risk of injury due to falling parts. Exercise extreme caution when handling the two components.

4 Function and design

4.1 multi EA 5100 basic module

The multi EA 5100 analysis system is modular and can be adapted to each measuring tasks by combining it with different detectors and sampling modules. The analysis system is used to determine the sulfur, nitrogen, chlorine and carbon content in solid, liquid, paste-like, viscous and gaseous samples. It can additionally analyze the AOX, EOX, EC/OC or TOC, NPOC and TIC sum parameters.

The core of the analyzer system is the multi EA 5100 basic module, which performs sample gas digestion and measuring gas drying. (As an exception, the measuring gas is dried in the detection module for chlorine determination.)

The analysis system is controlled and the measurement data is evaluated via the multiWin control and analysis software.

A self-check system (SCS) is integrated into the analysis system. The SCS consists of a combination of hardware components and software functions that automatically ensure proper function of the entire analysis system. Depending on the system setup, the SCS checks the important device safety and analysis quality parameters (e.g., gas flow, temperatures, pressures, system integrity, baseline stability, signal drift, cooling time, flame value, etc.) multiple times per second

4.1.1 Principle of operation

4.1.1.1 Vertical and horizontal operation

The basic module is equipped with a double furnace. This innovative technology enables operation of the combustion furnace in vertical or horizontal mode. Switching between the two operation modes is simple, and is performed by the user.

Vertical operation mode

In vertical operation mode, the sample aliquot is injected directly into the multi-purpose combustion tube via the injection port. The sampling module required is a multi-matrix autosampler, an auto-injector, a GSS module or an LPG module.

Advantages of vertical operation mode:
- For gases, LPG, liquid samples with normal viscosity
- Best for N, A and Cl trace and ultratrace analyses
- Fast analysis
- Small bench space required

Horizontal operation mode

In horizontal operation, solids and liquids are transferred to the multi-purpose combustion furnace on boats with Automatic Boat Drive (ABD). Sample feeding can be automated with an autosampler in combination with the ABD. Liquids can alternatively be transferred into the horizontal combustion furnace via the auto-injector.

Gases and LPG are injected directly via the injection port of the sample port of the ABD.

Advantages of horizontal operation mode:
- For gases, LPG, solids, liquid samples independent of viscosity
- Best for volatile liquids

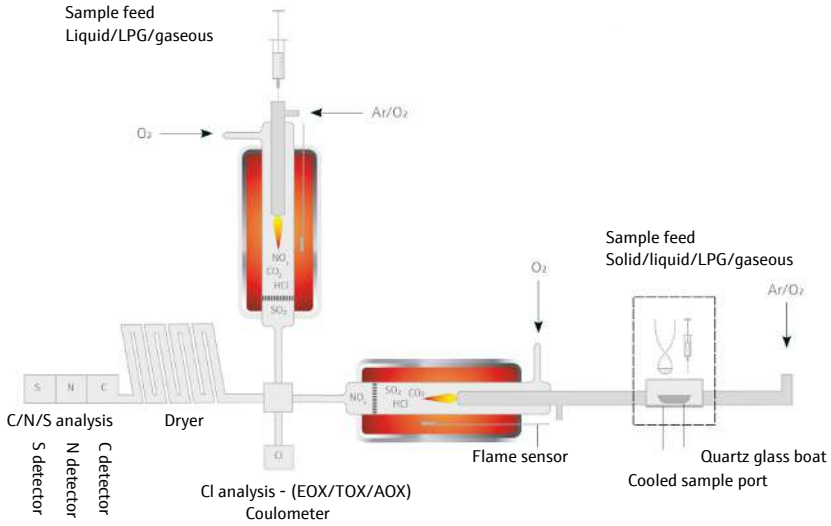


Fig. 1 Operating modes of the multi EA 5100

4.1.1.2 Sample Digestion

TS, TN, TC, TX and EOX

Sample digestion to determine TS, TN, TC, TX and EOX can be performed in vertical and horizontal operation mode.

Digestion is a two-stage process at 1000 to 1100 °C via pyrolysis with subsequent thermal oxidation. In the first phase, the sample components are pyrolyzed in an argon flow, and the generated pyrolysis gases are incinerated in an oxygen flow. Next, the remaining pyrolysis products are re-incinerated in a pure oxygen flow during the second phase.

This digestion is summed up via the following equations:

$R^*\text{-}H + O_2 \rightarrow CO_2 + H_2O$

$R^*\text{-}N + O_2 \rightarrow NO_x + CO_2 + H_2O$

$R^*\text{-}S + O_2 \rightarrow SO_2 + CO_2 + H_2O$

$R^*\text{-}X^{**} + O_2 \rightarrow HX^{**} + CO_2 + H_2O$

R* - carbonic substance

X** - F^-, Cl^-, Br^-, I^-

TOC

Digestion for TOC determination is performed in vertical operation mode via thermal oxidation in an oxygen flow at 700 °C. The aqueous samples are injected directly into the TOC combustion tube via the injection port.

TIC

Digestion for TIC determination is performed in the TOC module via wet chemical oxidation with phosphoric acid. The aqueous samples are injected into the TIC reactor manually.

AOX

Digestion for AOX determination is performed in the horizontal operation mode. The loaded active carbon is combusted in the oxygen flow at at least 950 °C and forms hydrogen halogen, carbon dioxide and water. The loaded active carbon is transferred to the multi-purpose combustion tube with or without a quartz container in the quartz glass boat with ABD.

EC/OC

Digestion for EC/OC determination is performed in horizontal operation mode. Digestion is performed in two phases. In the first process phase, the OC content adsorbed on the filter samples is thermally desorbed in a pure argon flow. The gaseous products are then re-incinerated in oxygen. In the second process phase, the remaining EC proportion in pure oxygen is converted completely to CO_2.

The filter samples are transferred to the combustion furnace via the ABD and a special feed program.

4.1.1.3 Measuring gas dryer

After leaving the combustion tube, the mixture is dried before it is transferred to the detectors.

Parameter	Method
TS. TN. TC. EC/OC	Membrane dryer (in the basic module)
TX. AOX. EOX	Concentrated sulfuric acid (in the CI module 5100)
TOC. NPOC. TIC	Condensation through Peltier cooling (in the TOC module 5100)

4.1.2 Design of the basic module

4.1.2.1 Main components

Main components of the basic module

The multi EA 5100 basic module contains the following main components:

- Electronics/internal device control
- Gas supply
- Combustion system
- Measuring gas transfer

All basic module components that must be operated or maintained by the user can be accessed via the 2 doors on the front side or via the removable side panels.

Electrical connection and gas connections) and the interfaces for connecting the system components are on the rear of the device.

Double furnace device

- Vertical and horizontal operating mode possible
- Opening with maintenance flaps at the right-hand side panel

Vertical operation

- Furnace in vertical installation position
- Right side panel closed

Fig. 2 Front view in vertical operation mode

1 Ventilator
2 Control electronics
3 Membrane dryer unit
4 Tilt device
5 Pump connection for N/S/C mode
6 Combustion furnace
7 Knob for tilting the furnace

Horizontal operation

- Furnace in horizontal installation position
- Opening with maintenance flaps at the right-hand side panel

Fig. 3 Basic module in horizontal operation mode

1 Control electronics
2 Combustion furnace
3 Auto-protection valve assembly
4 Gas box

4.1.2.2 Electrical components, display elements and connections

Internal device control

The control electronics are found on the rear of the basic module behind the panel when viewed from the front. The control electronics provides the power supply and control of the individual components and the communication with the control PC and other connected system modules.

Operational LED

Fig. 4 Basic module with a sampling module and a detection module.

A green LED is installed on the left door of the basic module After loading the control and analysis software, the LED lights up, indicating that the device is ready for operation.

Power switch, ports, connections

The power switch and the interfaces for connecting the system modules and for connecting the control PC can be found on the rear of the device.

The control PC can be connected via a USB port. The interfaces for connecting the sampling modules and for connecting the detectors are RS 232 interfaces.

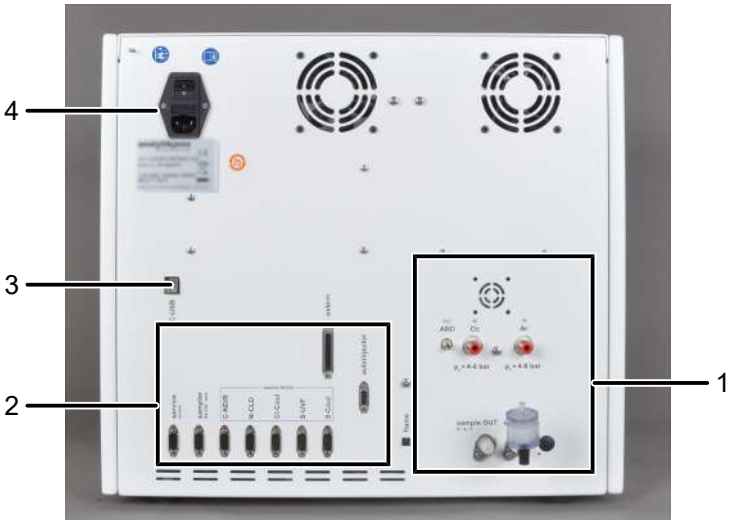


Fig. 5 Interfaces on the rear of the device

1 Gas connectors
2 Power connection, power switch
3 USB port for PC
4 Interfaces for detector modules and sampling modules

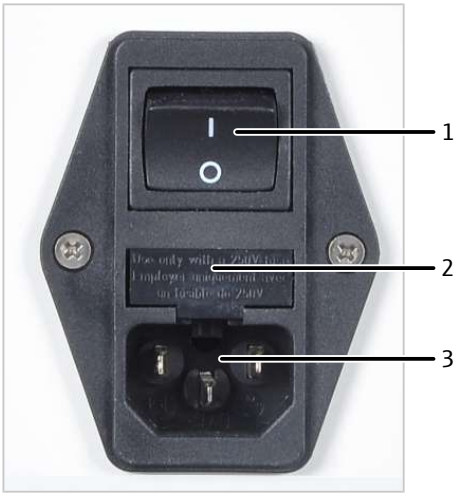


Fig. 6 Power connection, power switch

1 Power switch
2 Equipment fuse
3 Power connection

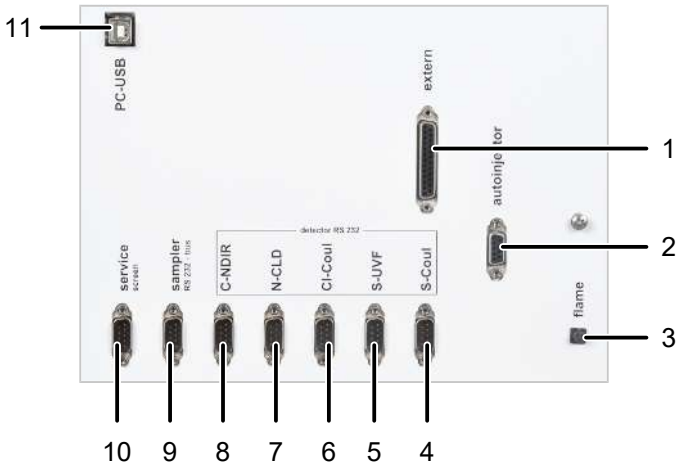


Fig. 7 Interfaces for detector modules and sampling modules

1 "External" connection
2 Autoinjector connection
3 "Flame" flame sensor connection
4 S-coulometer connection
5 S-UVF connection
6 CI-coulometer connection
7 N-CLD connection
8 C-NDIR connection
9 Sampler connection (RS-232 bus)
10 Service connection
11 Control PC connection

Interfaces in the device

The electrical connections for the combustion furnace, the flame sensor and the temperature sensor can be found on the rear interior side of the device. The connections are only accessible in the vertical installation position of the combustion furnace.

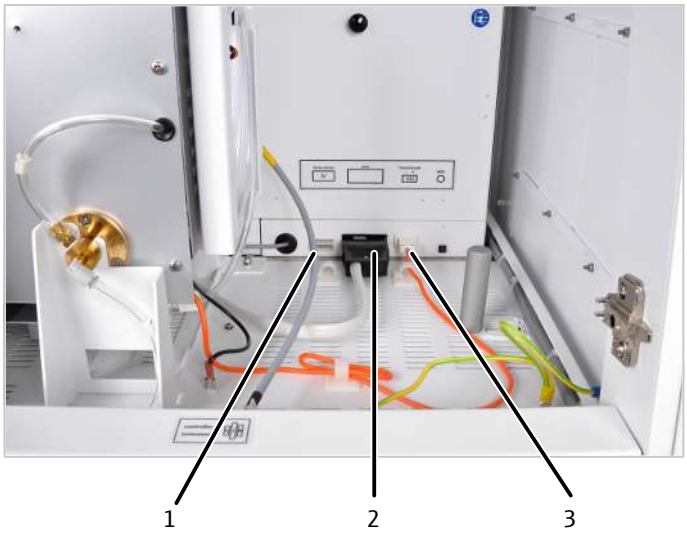


Fig. 8 Connection for the sensors and the combustion furnace in the device

1 Temperature sensor
2 Combustion furnace
3 flame sensor

The connections for the auto-protect valve assembly and the heated transfer line (only if the CI module is connected) can be found in the frame behind the door. The toggle switch for opening and closing the pneumatic seal in the auto-protection valve assembly is also located there.

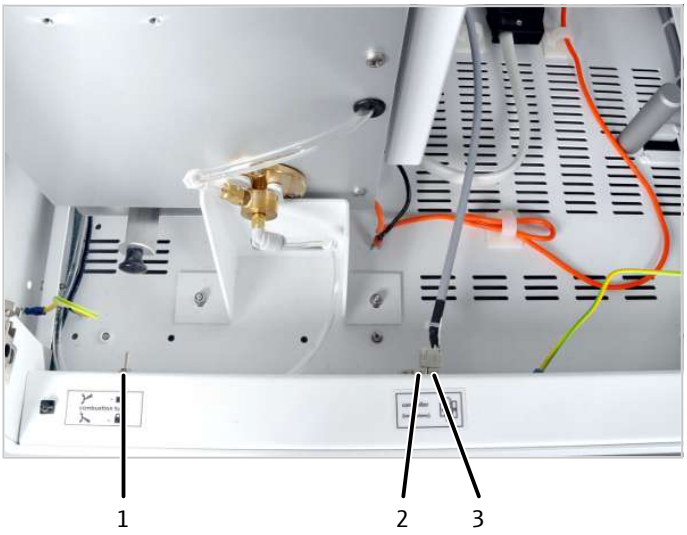


Fig. 9 Connections for the auto-protection valve assembly and transfer line

1 Toggle switch for opening and closing the pneumatic seal in the auto-protection valve assembly
2 Transfer line heating connection
3 Valve assembly connection

4.1.2.3 Gas supply/hose diagrams

Hose diagrams

The connection between the individual components is made with labeled hoses. The numbers circled in the hose diagram match the labels on the hoses in the multi EA 5100.

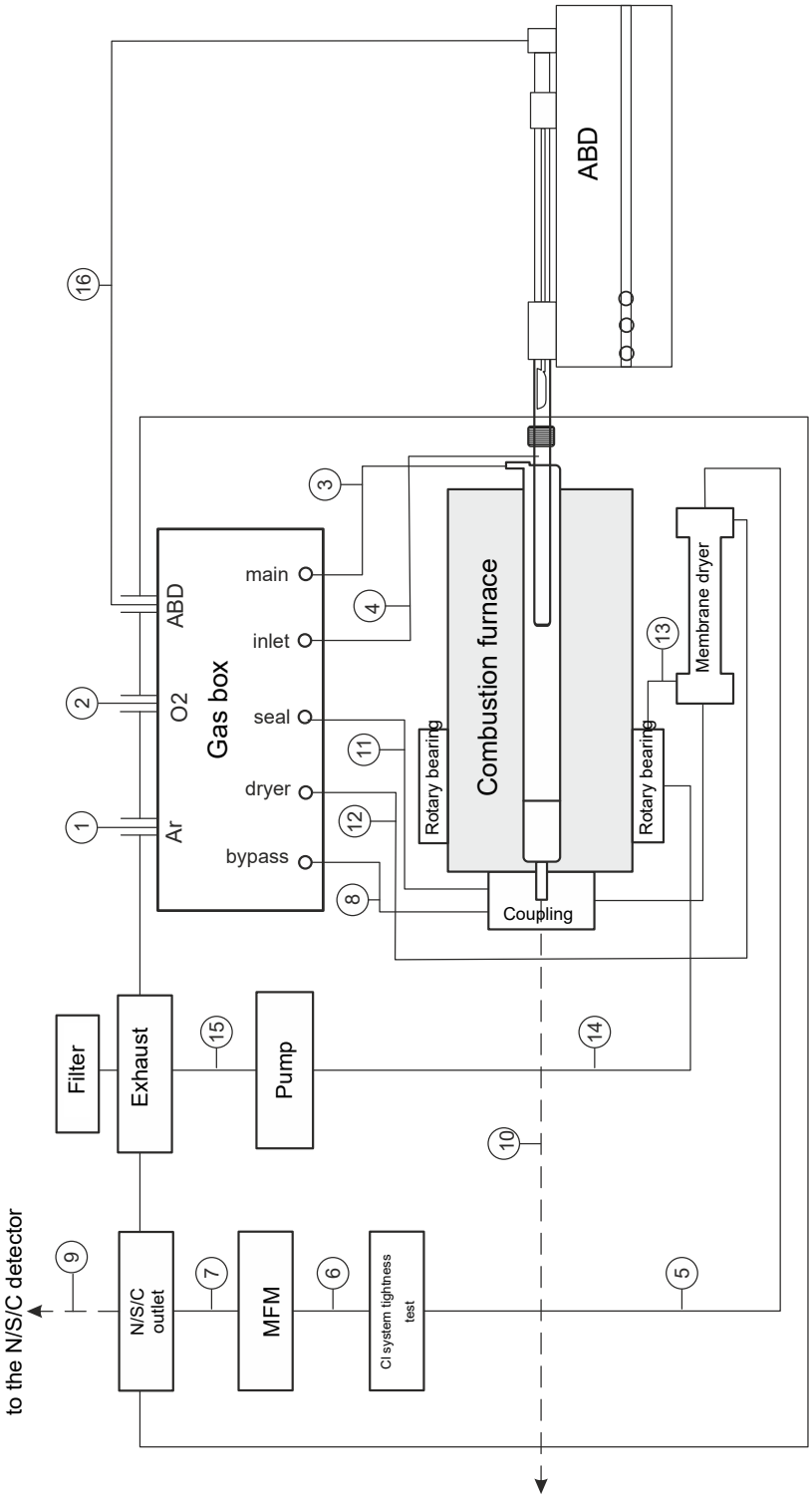


Fig. 10 Hose diagram for horizontal operation

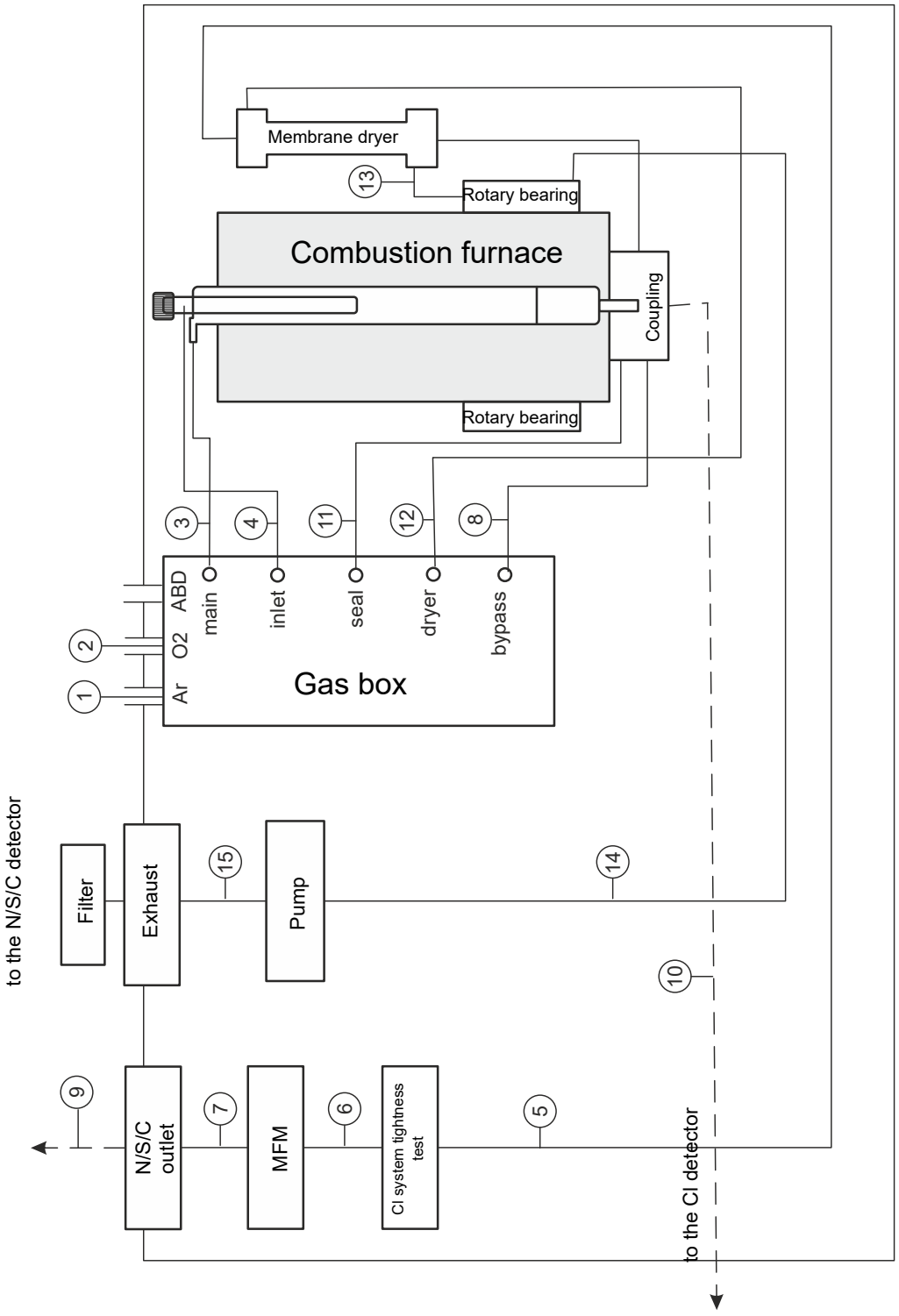


Fig. 11 Hose diagram for vertical operation

Gas connections at the rear of the device

The gas connections are located on the rear of the device. The gas supplies for oxygen and argon must be connected to the "IN O_2" or "IN Ar" connection via the included connection hoses (AD 6 mm, ID 4 mm.

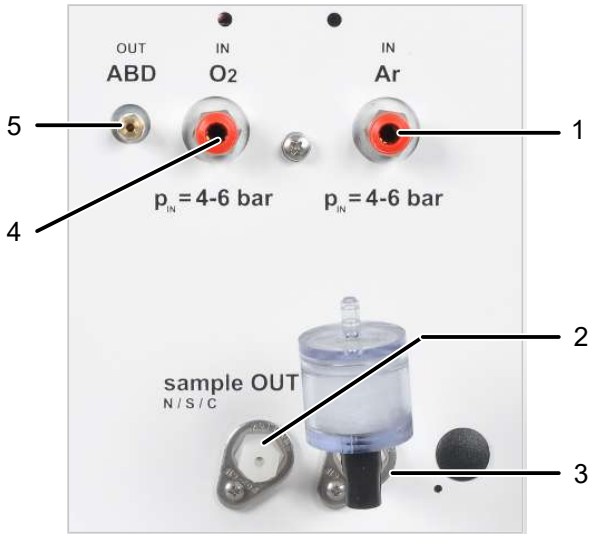


Fig. 12 Gas connections at the rear of the device

1	Argon gas inlet (Ar)	2	Gas outlet to the N/S/C module
3	Purge gas outlet of the membrane dryer with "exhaust" filter	4	Oxygen gas inlet (O_2)
5	Gas outlet to the ABD		

Gas connections on the gas box

The two process gases, argon and oxygen, are controlled via the gas box in the basic module. The gas box is located on the left side of the device.

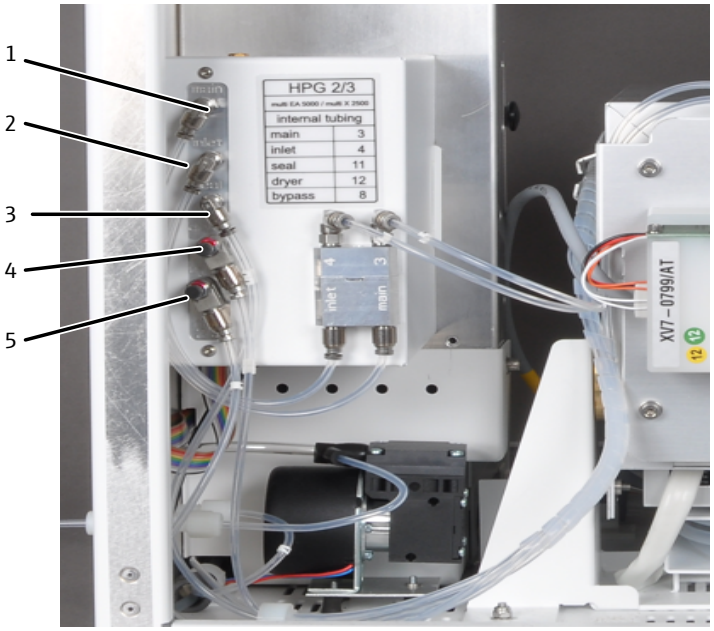


Fig. 13 Gas connections on the gas box

1	"main" – oxygen supply to the combustion tube (hose 3)	2	"inlet" – argon supply to the combustion tube (hose 4)
3	"seal" – argon connection for the seal of the auto-protection valve assembly (hose 11)	4	"dryer" – dry gas flow (oxygen) for the membrane dryer (hose 12)
5	"bypass" – argon connection for the safety purging (safety bypass) of the chlorine branch (hose 8)		

Connections on the combustion tube

Fig. 14 Connections on the combustion tube

1	Flame sensor connection (only if ABD is connected in horizontal mode)
2	Ar hose 4 connection (in horizontal operation mode, no argon is supplied here, argon is supplied via ABD)
3	O_2 hose 3 connection

Gas supply control

The composition of the gas mixture for optimum gas digestion is controlled via the flow management system (FMS).

4.1.2.4 Combustion system

A resistance-heated combustion furnace for digestion temperatures of 700 to 1100 °C is used in the basic module. Digestion with the multi-purpose combustion tube is performed at temperatures of 950 °C or 1000 to 1100 °C, depending on the application. The double furnace device can be switched to the required operating mode quickly via the tilt device.

Fig. 15 Combustion furnace in the vertical and horizontal operating mode

A multi-purpose combustion tube used for all standard applications, both in vertical or horizontal operation mode, is installed in the combustion furnace. The combustion tube is made of quartz glass. The auto-protection assembly group connects the combustion tube to the measuring gas dryer or the further path of the measuring gas.

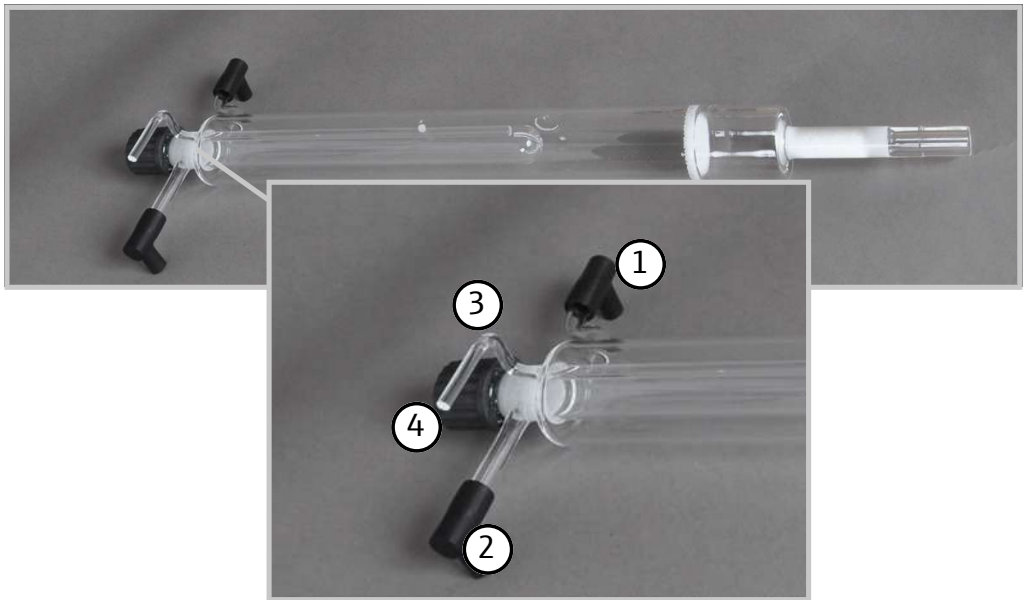


Fig. 16 Multi-purpose combustion tube

1 Oxygen supply connection
2 Argon supply connection
3 Flame sensor connection
4 Screw cap with septum (only for vertical operation mode and operation with an auto-injector)

4.1.2.5 Measuring gas dryer

Measuring gas is dried in accordance with the measuring method:

- To determine TS. TN. TC. EC/OC via membrane dryer:
  The membrane dryer is mounted on the furnace. To increase drying effectiveness, purge gas (O_2) is drawn through the membrane dryer with a pump. The membrane dryer is maintenance-free.

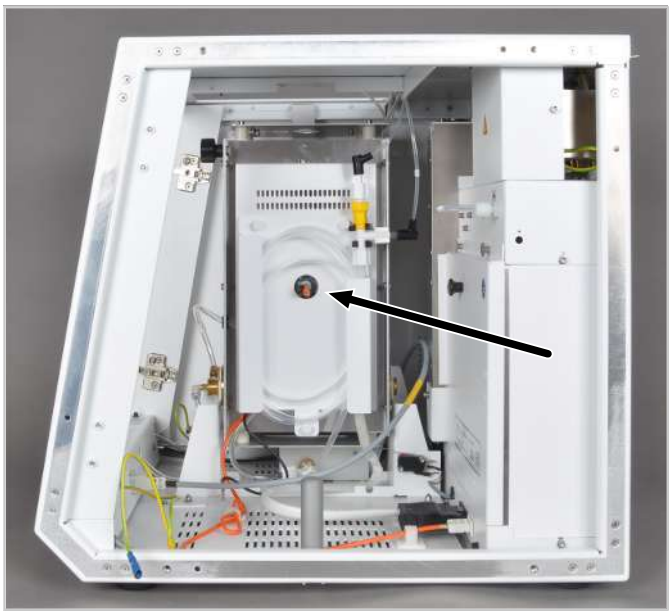


Fig. 17 Membrane dryer

- To determine TX. AOX. EOX via concentrated sulfuric acid:
  Sulfuric acid is dried in the Cl module 5100. The measuring gas is routed to the sulfuric acid container via a transfer line.
- To determine TOC. NPOC. TIC via condensation with Peltier cooling in the TOC module 5100.

4.1.2.6 Type plate

The type plate is located on the rear of the basic module and system components.

The type plate contains the following information:

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

4.2 Sampling modules

4.2.1 Auto-injector

There are two types of auto-injector. The classic auto-injector is suitable for vertical and horizontal operation mode, while the Autoinjector AI-EA is only used in vertical operation mode.

The auto-injectors are suitable for the following applications:

- In vertical operation mode for non-viscous liquids and colorless EOX extracts

- In horizontal operation mode only for volatile liquids

The auto-injectors are **unsuitable** for the following sample types:

- Viscous liquids and their solutions
- Solids suspended in solution
- Colored EOX extracts
- Water analyses (TC/NPOC determination)

5 Installation and commissioning

5.1 Installation conditions

5.2 Installation location requirements

Ambient conditions

The climate conditions for the installation location are listed in the technical data (→ "Technical data for the multi EA 5100 " 🖹 171). If required, ensure that the room is temperature-controlled.

Laboratory conditions

The device is only approved for indoor use. The installation location should have the characteristics of a chemical laboratory. It must meet the following conditions:

- Atmosphere free from hydrocarbons, halogens, sulfur compounds and nitrogen oxides
- Atmosphere with low dust levels
- No vibrations
- No smoking in the operating room of the device

Installation location requirements

The requirements for the installation location of the device are as follows:

- No caustic vapor in the immediate vicinity of the device and its system components. These could corrode the device connections and modules.
- Free from draft air; do not install the device close to windows or doors
- Away from electromagnetic sources of interference
- No direct sunlight and away from radiant heaters
- The front door and air vents must not be obstructed by other equipment or furnishings
- Maintain a safety distance of at least 20 cm from the rear of the device to other devices or walls.

5.3 Power supply

WARNING

Danger due to electrical voltage

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

The device operates on single-phase alternating current.

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

The electrical connection data can be found in the technical data (→ "Technical data for the multi EA 5100 " 🖹 171).

5.4 Gas supply

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

The connection hoses with outer diameter (AD) 6 mm and inner diameter (ID) 4 mm are provided with the device. Their lengths are 2 m. If other lengths are preferred, please contact the Analytik Jena GmbH+Co. KG customer service department. The required gases and their qualities are listed in the technical data (→ "Technical data for the multi EA 5100 " 171).

5.5 Device layout and space requirements

The space required for the modular analysis system depends on all the components that make up the measuring station. The analysis station always includes:

- Basic module
- 1 sampling module (to the right of the basic device or on top of it)
- 1 detection module (to the left of the basic device)

Several modules may also be placed in a row.

Component	Width x Height x Depth [mm]	Mass [kg]	Arrangement
Basic module multi EA 5100	510 x 470 x 550 mm	25 kg	
Detection modules			
N module 5100	300 x 500 x 550 mm	13 kg	As last detector in a row
S module 5100 basic S module 5100 MPO	300 x 470 x 550 mm	13 kg	Left/right of other detectors
Cl module 5100	300 x 470 x 530 mm	12 kg	To the immediate left of the basic module
S module 5100 coulometric	300 x 470 x 530 mm	11 kg	Left of the Cl module 5100
C module 5100	300 x 470 x 530 mm	12 kg	Left/right of other detectors
TOC module 5100	300 x 470 x 530 mm	12 kg	To the immediate left of the basic module or to the left of the Cl module 5100
Sampling modules			
ABD	520 x 210 x 500 mm	12 kg	To the right of the basic module
MBD	500 x 80 x 80 mm	0.35 kg	To the right of the basic module
Multi Matrix Sampler	ca. 510 x 500 x 410 mm	ca. 9.5 kg	On the basic module or on the ABD*
Autoinjector (without syringe, Ø x L)	30 x 80 mm 80 x 110 mm	0.5 kg	Installed on the basic module, or on the right of the basic module

Component	Width x Height x Depth [mm]	Mass [kg]	Arrangement
Auto injector coupling (Ø x L)			
Autoinjector AI-EA	150 x 270 x 240 mm	1.5 kg	On the basic module
GSS/LPG combi module, GSS module or LPG module 2.0	300 x 800 x 550 mm	11 kg/ 12 kg	To the right of the basic module or ABD

* For the temperature-controlled autosampler MMS-T and the Liquids kit TMP: There is sufficient space provided for the inclusion of a thermostat (approx. 250 x 650 x 400 mm).

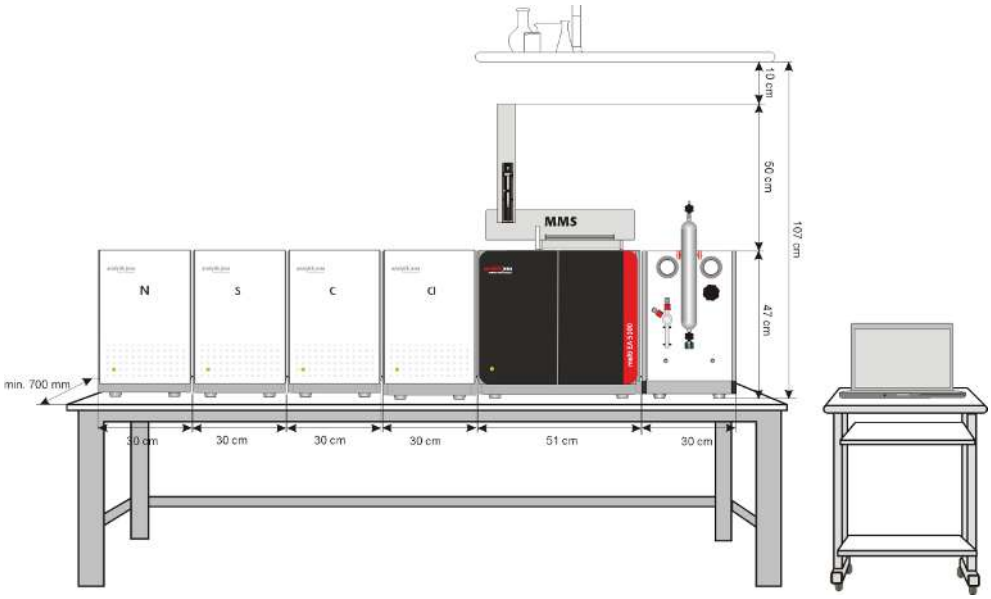


Fig. 18 Space required for the basic device and modules (vertical operation)

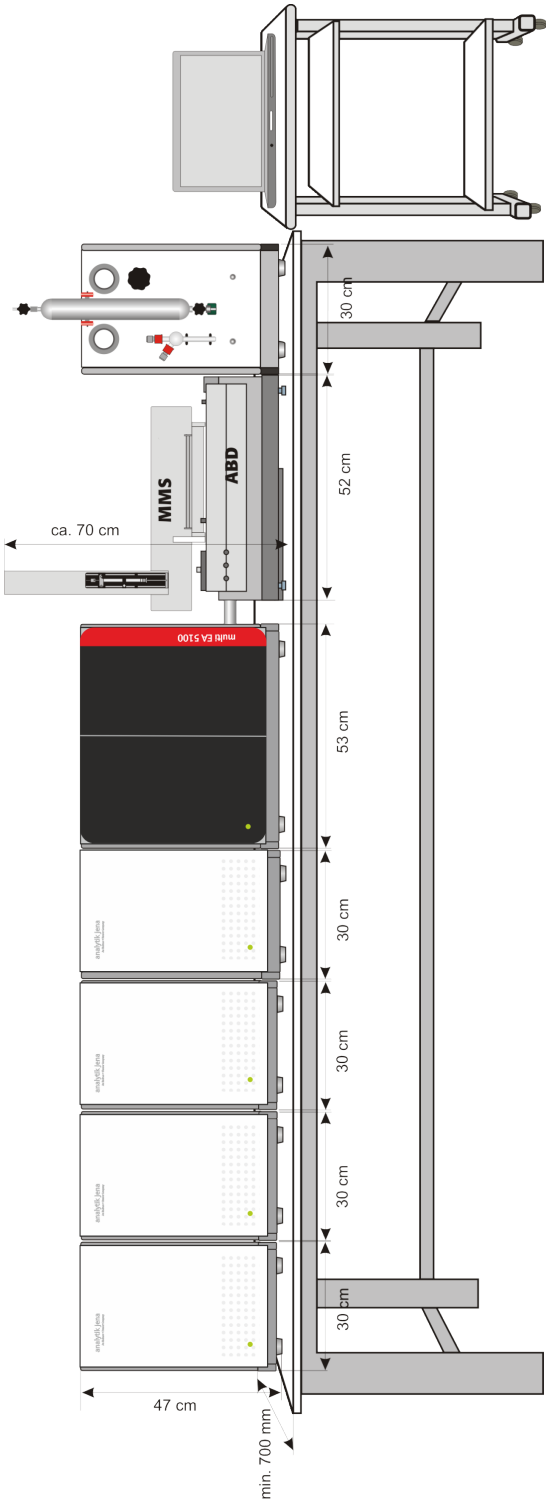


Fig. 19 Space required for the basic device and modules (horizontal operation)

5.6 Installing and commissioning the analysis system

WARNING

Danger due to incorrect commissioning

- The analysis system may only be set up, mounted and installed by the Analytik Jena GmbH+Co. KG customer service department.
- Any unauthorized access of the device can endanger the user and the operational safety of the equipment and limits or completely voids any warranty claims.

NOTICE

Retain the transport packaging

Return transport for maintenance must be in the original packaging. This alone prevents transport damage.

The basic module, the sampling modules and the detectors are unpacked and assembled by the Analytik Jena GmbH+Co. KG customer service department or personnel trained and authorized by them.

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

The customer service department tests the function of the analysis system after installation and documents the test.

Installing the basic module

- Carefully remove the basic module and its components from the transport packaging. Do not damage the transport packaging!
- Place the basic module at its intended location.
- Ensure adequate space for the additional system components (sampling modules, detectors).
- Install the combustion furnace (→ "Removing and installing the combustion furnace" 🖹 139).
- Install the auto-protection valve assembly (→ "Maintenance of the auto-protection valve assembly" 🖹 129).
- Install the combustion tube in the combustion furnace (→ "Maintenance of the multi-purpose combustion tube" 🖹 124).
- Place the additional system modules on their intended locations and connect them.

CAUTION

Danger of short circuit!

- Only connect the basic module and other system components when they are powered down and switched off.
- Before connecting the power supply cable, set the power switch of the rear of the device to "0".
- Only use the IEC connection cable included in the scope of delivery for the connection to the power supply (VDE label, 1.5 m long). Extensions of the supply cable are not permitted!

NOTICE

Settled condensation and temperature differences can damage individual components of the basic module during commissioning.

- If there is any temperature difference between the storage location and the operating location, allow the analyzer system to acclimatize for at least one hour in the room it will operate in.

Connecting power and gases

- Connect the power cable to the connection of the rear of the basic module.
- Connect the power plug to a grounded power outlet.
- Connect the supplied connection hoses to the pressure reducers of the gas supply and the the O_2 and Ar gas connections on the rear of the device (media connections of the rear of the device).
- Set the inlet pressure on the pressure reducers (600 kPa (6 bar)).
- Connect the computer and connect it to the basic module via the supplied USB cable.
- Connect further system components (detectors, sampling modules) to the basic module.

✓ The basic module is now ready for operation and can be switched on.

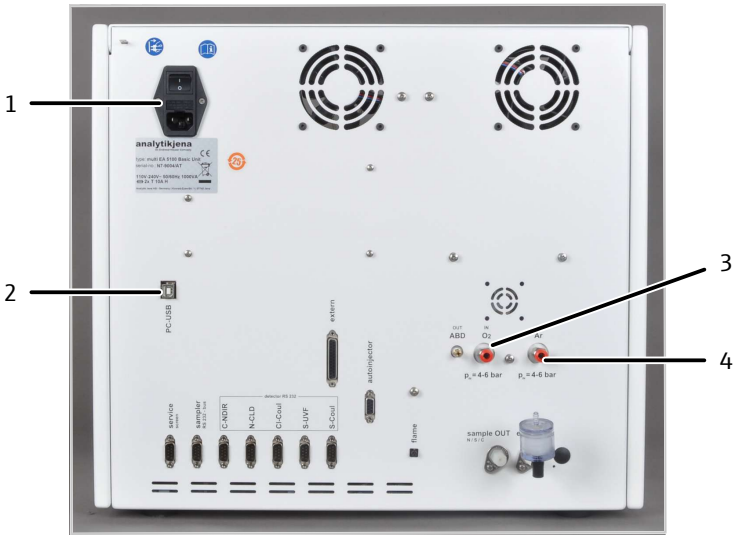


Fig. 20 Media connections of the rear of the device

1 Power connection
2 USB port for computer
3 "O_2" oxygen connection
4 "Ar" argon connection

6 Operation

6.1 General notes for measuring operations

Only use the correct sampling modules for the corresponding matrix and installation position of the furnace for sampling.

Furnace installation position	Sample type	Sampling
Vertical:	Liquids	■ Autoinjector ■ Multi Matrix Sampler MMS
	Gas, non-pressurized	■ GSS module
	Gas, pressurized	■ GSS/LPG combi module ■ GSS module with GSS adapterbox
	LPG	■ LPG module 2.0 ■ GSS/LPG combi module
Horizontal:	Solids	■ Automatic Boat Drive ABD ■ ABD with MMS
	Liquids	■ ABD with manual dosing syringe ■ ABD with MMS ■ Autoinjector
	Gas, non-pressurized	■ GSS module
	Gas, pressurized	■ GSS/LPG combi module ■ GSS module with GSS adapterbox
	LPG	■ LPG module 2.0 ■ GSS/LPG combi module

Observe the following during analysis:

- To add samples of flammable substances in horizontal operation, always use ABD with a flame sensor in automatic or automatic plus mode or an Autoinjector.
- To add samples of flammable substances, do not use the ABD in parameter mode, or only use it with program parameters designed and tested for it (risk of soot!).
- Do not exceed the maximum permissible sample amounts (→ "Technical data for the multi EA 5100 " 🖹 171).
- Adjust the dosing rate to the sample matrix and observe the maximum dosing speeds (→ "Technical data for the multi EA 5100 " 🖹 171).
- Standard solutions with organic solvents can change composition rapidly due to the volatility. Therefore, ensure that the clear space above the liquid in the sample cup is small when preparing and storing samples. Store the solutions in the refrigerator. The boiling points of the materials used should also not differ by more than 50 °C.
- Adjust the sample volume to the expected concentration to remain within the measuring range of the detector.
- Begin the analysis with a standard solution and determine the daily factor. If the value measured for the standard solution differs by more than 20 % from the target value, repeat the measurement. If required, look for the source of the fault. If required, re-calibrate the analysis system.
- For examining very low element contents, examining a blind value is recommended before the daily factor. The blind value measurement cleans the analysis system.

6.2 Selecting the measurement method

Select the proper measurement method for each sample with the following table.

Some parameters can only be analyzed with horizontally or vertically installed furnaces
- Horizontal: AOX, EC/OC
- Vertical: TOC, TIC, NPOC (in water)

For other samples, the recommended measurement method depends on the nature of the sample.

Sample	Furnace installation position and sampling	Comment
TS, TN, TX, TC in:		
Organic solids, e.g., wax, polymer	Horizontal for ABD ABD with MMS	ABD with flame sensor For trace analysis: Use 1 quartz glass boat for all samples MMS with temperature-control unit in heating mode for high-viscosity liquids ABD without MMS for non-homogeneous samples
High-viscosity organic liquids, gels and paste-like samples, e.g., crude oil, asphalt, bitumen, tar		
Bio-Diesel		
Oils such as crude oil, paraffin oil and vegetable oil		
Volatile liquids such as petroleum ether, methanol, naphtha		
Liquids or solids with high element content (> 100 mg/l)		
Organic liquids with normal viscosity, such as fuels, solvents	Horizontal for Autoinjector ABD ABD with Multi Matrix Sampler	ABD with flame sensor
	Vertical for Autoinjector and Autoinjector AI-EA MMS	For trace analysis: must be vertical
EOX	Horizontal for ABD ABD with MMS	ABD with flame sensor Sample (colorless, colored) in n-hexane or petroleum ether
	Vertical for MMS Autoinjector	Only colorless samples in n-hexane
AOX	Horizontal for ABD ABD with MMS	Column method: Activated carbon in a quartz container in a quartz glass boat Batch method: Activated carbon with no quartz container in a quartz glass boat with holding-down clamp

Sample	Furnace installation position and sampling	Comment
EC/OC	Horizontal for ABD ABD with MMS	Filter in a quartz glass boat with hold-down clamp
TC, TOC, NPOC in water	Vertical for Multi Matrix Sampler or manual sampling	
TIC in water	Vertical (manual sampling)	Direct injection in the TIC reactor
Gas, non-pressurized	Horizontal or vertical for GSS module with ABD (horizontal)	For trace analysis: Vertical + GSS module recommended
Gas, pressurized	Horizontal or vertical for GSS/LPG combi module GSS module with GSS adapterbox with ABD (horizontal)	
LPG	Horizontal or vertical GSS/LPG combi module LPG module 2.0 with ABD (horizontal)	For trace analysis: Vertical + GSS/LPG combi module recommended

6.3 Switching on the basic module and the modules

Before switching on the basic module, always check:

- The other components (detection modules, sampling modules, PC) are connected to the basic module.
- The gas supply is connected in accordance with instructions. The inlet pressure is exactly 600 kPa (6 bar).
- The samples have been prepared.

Switch on the basic module as follows:

- Open the valves at the pressure reducers of the gas supply.
- Switch on all required components (detection modules, sampling modules, PC).
- Switch on the basic module via the power switch.
- The basic module boots up. The LED on the front lights up in green after approx 30 s.
- Start the multiWin program. Log in with user name and password.
- Click the **[Initialize analyzer]** button.
  - ✓ Initialization takes place after successful login.

NOTICE

Observe the run-in period

In the **Status analyzer** window, components that are not yet ready for operation are displayed in red. The heating time for 1050 °C is approx. 30 min. Measurement is not possible during the run-in period.

- If the analyzer is still not ready after approx. 30 min, perform a fault search (→ "Troubleshooting" 🖹 98).

6.4 Switching off the basic module and the modules

NOTICE

Risk of overheating

If the basic module is switched off too early, the electronics can overheat and become damaged due to lack of cooling.

- Only switch off the basic module after a cooling time of 1 h.

Switch off the basic modules and its modules as follows:

- Exit the multiWin program.
- Switch off the connected modules via their respective power switches.
- Only switch off the basic module after a cooling time of 1 h.
  If the system is switched off too early, the electronics inside the device can overheat due to lack of cooling.
- If a Cl module 5100 is connected: Before closing the gas supply, remove the sulfuric acid container from the module and completely drain the sulfuric acid.
- If a S module 5100 coulometric is connected: Before closing the gas supply, remove the gas transfer line from the gas inlet tube of the measuring cell.
- After switching off the modules, close the gas supply.
- Exit the Windows operating system and switch off the PC.

  ✓ Shutdown of the basic modules and its modules is complete.

6.5 Recommissioning after emergency shutdown (Cl module 5100)

WARNING

Chemical burns due to concentrated sulfuric acid

If a Cl module 5100 is connected to the basic module, sulfuric acid can still be in the gas transfer line and the auto-protection valve assembly after an emergency shutdown.

- Wear protective clothing when working on the sulfuric acid container.
- Exercise particular caution when checking the gas transfer line and the auto-protection valve assembly.
- Observe all specifications in the safety datasheet.

CAUTION

Risk of burns from the hot furnace and from the gas transfer line

- Allow the device to cool before recommissioning.

Observe the following when recommissioning the basic module and Cl module 5100. Vent the analysis system after an overpressure fault (fault message"206 – gas pressure fault") as described in the following:

- Carefully disconnect the measuring gas hose from the measurement cell.
- Carefully disconnect the gas transfer line from the sulfuric acid container and remove the sulfuric acid container from the module.
- Disconnect the gas transfer line of the auto-protection valve assembly in the basic module. Remove the heating cable plug from its socket.
- Carefully remove the gas transfer line and check it for contamination with sulfuric acid.
- If necessary, clean the gas transfer line:
  - Rinse the gas transfer line with distilled water and then with ethanol.
  - Dry the gas transfer line (e.g. by blowing it through with an inert gas).
- Wait for the pressure in the system to decrease. Then switch off the basic module. Shut off the gas supply.
- Open the seal for the auto-protection valve assembly in the basic module. To do this, flip the toggle switch up. Remove the valve assembly plug from its socket.
- Carefully remove the auto-protection valve assembly from the basic module and check it for contamination with sulfuric acid.
- If necessary, clean and dry the assembly. Replace the filter.
  If cleaning is not possible or if the auto-protection valve assembly is damaged, it must be replaced prior to recommissioning.
- Reinstall the auto-protection valve assembly in the basic module. Connect the assembly via the cable. Ensure that a filter has been inserted in the auto-protect valve assembly.
- Refill the sulfuric acid container with sulfuric acid and insert it in the detection module. Connect the measuring gas hose with the sulfuric acid container.
- Refit the gas transfer line:
  - Connect the gas transfer line with the auto-protection valve assembly. Plug the heating cable plug into its socket.
  - Route the gas transfer line through the wall of the basic module top the detection module. Connect the gas transfer line to the sulfuric acid container.

  ✓ The basic module and the detection module can be switched back on again.

7 Nitrogen analysis with the N module 5100

7.1 Function and design

7.1.1 Function and measuring principle

Expansion of the basic module with the detection module allows the determination of the nitrogen content in solids, liquids, and gases via chemiluminescence.

Organic nitrogen compounds can be determined as a TN sum parameter with the analysis system. Inorganic nitrogen compounds are only detected if they can be digested in the combustion furnace. Pure nitrogen cannot be analyzed.

The chemiluminescence of the reaction between nitrogen monoxide (NO) and ozone (O_3) is used for the determination. This reaction creates nitrogen dioxide in its excited state (NO_2*) for a short time. During the transition to the base state, the nitrogen dioxide emits electromagnetic radiation in the visible light spectrum. The light emitted is proportional to the NO_2* concentration. This allows measurement of the concentration via light. NO is the only substance involved in the reaction, which means that this method is very selective and not influenced by any other constituents of the measuring gas.

$NO + O_3 \rightarrow NO_2^* + O_2$

$NO^* \rightarrow NO_2 + hv$

The measuring gas is created during incineration of organic nitrogen compounds in the basic module. It contains a mixture of NO and NO_2, generally known as NO_x.

$R\text{-}N + O_2 \rightarrow NO_x + CO_2 + H_2O$

R: Hydrocarbon residue, NO_x: Mixture of NO and NO_2

The measuring gas is passed through a converter which renders the NO_2 portion usable for the reaction and hence for detection purposes. The converter reduces NO_2 to NO.

The ozone required for the reaction is created in the device from the supplied oxygen (O_2). Excess O_3 is removed in the ozone decomposer after the reaction. The toxic gas is not released into the ambient air.

7.1.2 Design

The detection module is used to determine the nitrogen content via chemiluminescence. All components required for determination are mounted inside the sealed housing.

Fig. 21 Basic module with detection module and sampling module

The detection module consists of the following components:

Component	Function
Micro-plasma chamber	Preparation of ozone (O_3) from oxygen
Converter	Transformation of nitrogen dioxide (NO_2) into nitrogen monoxide (NO)
Reactor with sensor	Reaction of nitrogen monoxide (NO) with ozone (O_3) to produce nitrogen dioxide (NO_2*) Detection of the amount of emitted light
Chemical and thermal ozone decomposer	Decomposition of excess ozone (O_3)
Diaphragm pump	Conveyance of the measuring gas through the detector
Differential pressure sensor	Regulation of the pressure compensation between the variable measuring gas flow and the fixed suction flow of the membrane pump.
Absorber	Cleaning of the aspirated air before entering the diaphragm pump

7.1.3 Connection

The equipment door at the front is closed tightly and cannot be opened. An LED is installed on the door. The LED flashes during the run-in period of the detection module and lights up permanently when the module is operational.

The following connections can be found on the rear of the detection module:

- Main switch, power connection, device fuses
- Media connections for gases and waste
- Interface for connection to a basic module
- Service interface with programming button.

A diagram at the center on the rear of the device explains the different connections.

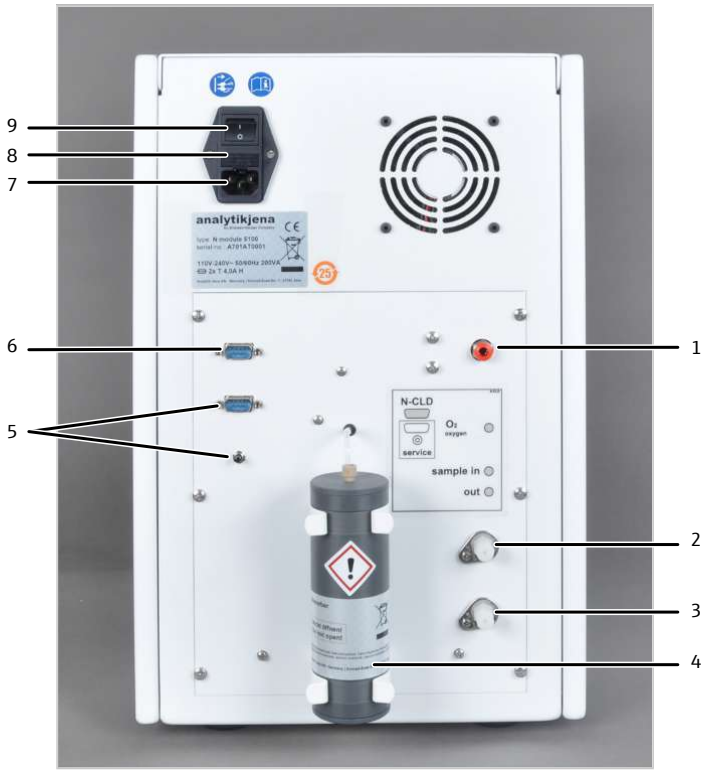


Fig. 22 Rear of the nitrogen detector

1 O_2 gas inlet
2 Measuring gas inlet
3 Measuring gas outlet
4 Absorber
5 Service interface, programming button
6 Interface to the basic module
7 Power connection
8 Fuse holder
9 Equipment switch

The device switch for switching the detection module on and off is located on the top right of the rear of the device (when viewed from the front). The device fuse and power connection are located beneath it.

Communication with the basic module is performed via a 9 pin interface cable. The interface of the rear of the detector is marked with "N-CLD".

The gas for producing ozone is connected to the "O_2/air" quick-release connector on the rear of the detector. The hose for the measuring gas coming from the basic module is connected to the "sample in" gas inlet.

In order to equalize pressure differences caused by differing gas flows, it is possible to use an absorber to allow air to enter the device. The absorber filters constituents from the air which may distort the analysis.

The "service" interface is for service purposes only (monitoring function only). The transmission data log of the detection module is output via this interface. A null modem cable is required to connect. The programming button is also only for service purposes (firmware updates).

7.2 Installation

NOTICE

Connecting or disconnecting electrical contacts may damage the sensitive electronic components of the basic module and of the detection module.

- Always connect the modules to power when they are switched off.

NOTICE

The detection module is equipped with a pump that can disrupt the function of other optical detection modules or lead to serious errors.

- Always connect the detection module as the last in any series of the detectors.

- Install the detection module as the last in any series of detectors.
- Install the absorber on the rear of the device:
  - Fasten the two holding clamps with the supplied screws.
  - First press the absorber into the upper holding clamp and then into the lower one.
  - Connect hose 6 to the absorber. Do not pull the hose out of the device!

Fig. 23 Absorber

1 Hose 6 connection
2 Holding clamp
3 Absorber

- Connect the supplied power cable to the power cable connection on the rear of the module and to an earthed socket. Observe the permissible maximum voltage!

- Insert the oxygen hose in the "O_2" connector.
  NOTICE! To release the hose, press the red ring into the connector and pull the hose out of the connection.
- Connect the detection module with the basic module via the interface:
  "N-CLD" interface on the rear of the detection module
  "N-CLD" on the rear of the basic module.
- Connect the measuring gas hose of the basic module to the "sample in" gas inlet on the rear of the module.
- Connect an approx. 50 cm long hose to the "out" connection. Route the hose away from the device.

  ✓ Connection of the detection module is complete.

The hose at the "out" connection prevents reaction gases from being sucked back into the device by the absorber, disrupting the analysis. The hose does not have to be connected to the laboratory exhaust unit.

Fig. 24 Connecting detection modules to the basic module

1 Detection module interfaces

2 Measuring gas outlet (for measuring nitrogen, sulfur, carbon)

7.3 Operation

CAUTION

Risk of respiratory problems due to leaking ozone

If the gas hoses have not been properly connected to the ozone generator, ozone may leak out of the detection module.

- If you can smell ozone, switch off the device and check the connection of the gas hoses on the ozone generator.

- Switch on the basic module and the detection module.
  - ✓ The devices boot up. The status LED on the front of the basic module light up in green after approx. 30 s.
  - ✓ The LED on the front of the detection module flashes during the run-in period. Depending on the detector, the run-in period can take up to 30 min. After this, the LED will light up continuously. Starting a measurement is only then possible.
- Open the gas supply and set the required gas pressure.
- Switch on the PC.
- Start the control and analysis software and login with your username and password.
- Initialize the analysis system by clicking on **[Initialize analyzer]**.
  - ✓ The initialization and automatic detection of all connected components will be carried out.
- Ready the samples.
- Activate a pre-existing method via the **Method** | **Method - activate** menu item.
- Alternatively: Create a new method in the **Method** | **Method - new** menu. Select the measurement parameter in the method. Release and activate the method.
- Select **Start** | **Start - Analysis** in the menu.
- Select an analysis group or create a new one and confirm via **[OK]**.
- Create an analysis sequence.
- Enter the sample ID for all sample in the **Name** field.
- Release all sequence lines
- Confirm the entries with **[OK]**.
- Click the **[Start Measurement]** button.
  - ✓ The prepared analysis sequence is processed.

For manual sampling, follow the instructions in the software.

8 Chlorine analysis with the Cl module 5100

8.1 Function and design

8.1.1 Function and measuring principle

Expansion of the basic module with the detection module allows determination of the chlorine content in solid, liquid, paste-like, viscous and gaseous samples. When doing so, the device will determine the bromine and iodine content as proportions of the total chlorine of the sample as well. Fluorine is not detected.

Organic halogen compounds can be determined as TX, AOX and EOX sum parameters with the analysis system. Inorganic halogen compounds are only determined if they can be digested via the combustion furnace. Pure halogens cannot be analyzed.

Organic halogen compounds are converted into hydrogen halides, carbon dioxide and water in the basic module.

The measuring gas flow is routed to the detection module via a transfer line and dried there. The hydrogen halides (HCl, HBr, HI) are determined there via microcoulometric titration. Hydrogen flouride (HF) is not determined.

In the first step, hydrogen chloride HCl* dissolves in the electrolyte, and dissociates to hydrogen and chloride ions (H+, Cl-). In the measuring cell, the chloride ions react with silver ions created via electrolysis to form silver chloride (AgCl). To achieve the most complete reaction to AgCl, titration is carried out in a strong acetic acid electrolyte. The solubility product of AgCl is reduced in the acetic acid electrolyte.

$R\text{-}X + O_2 \rightarrow HX + CO_2 + H_2O$

$HX^* \rightarrow H^+ + X^-$

$Ag \rightarrow Ag^+ + e^-$

$Ag^+ + X^- \rightarrow AgX$

R: Hydrocarbon residue, X: Cl, Br, I, * HBr und HI are determined as proportions of AgBr and AgI.

The end point of the titration is displayed potentiometrically. In accordance with Faraday's law, the amount of chloride ions can be calculated from the amount of charge expended to create the silver ions.

8.1.2 Design

The detection module consists of the following main components:

- Wide-range coulometer for amperometry and potentiometry
- Stirrer/cooling block for the measuring cell (with automatic cell detection)
- Measuring cells with electrodes
- Sulfuric acid container with safety attachment and gas inlet
- Connections, interfaces
- Electrode containers

Fig. 25 Design of the chlorine detector (without measuring cell)

1 Safety attachment
2 Sulfuric acid container
3 Stirrer/cooling block
4 Connections for electrodes, measuring cells

The wide-range coulometer has 3 application ranges. A special measuring cell is used for each application range:

- "high sensitive" for low chlorine content (e.g., fuels, LPG, EOX)
- "sensitive" for medium chlorine content (e.g., waste oil, AOX)
- "high concentration" for high chlorine content (e.g., waste, polymers, waste oil)

The measuring cells are automatically detected when they are inserted into the stirrer/cooling block. When the detection module is switched on, the magnetic stirring rod in the measuring cell starts moving. The preset cell temperature is 18 °C and can be modified as a method parameter in the control and analysis software.

The "sensitive" measuring cell

The "sensitive" measuring cell is used for chlorine contents between 1 to 100 µg.

The measuring cell consists of the electrode space, which holds the electrolyte solution, and the stirrer block in the detection module. A generator anode in the form of a stable silver plate (silver circle) is located on the bottom of the electrode space. The magnetic stirring rod runs above the anode.

The cell is sealed airtight with a lid and three knurled head screws. The lid has two openings:

- The opening marked "electrode" is for the amperometric combined electrode.
- The unmarked opening is used for direct injection into the measuring cell or to connect to the exhaust.

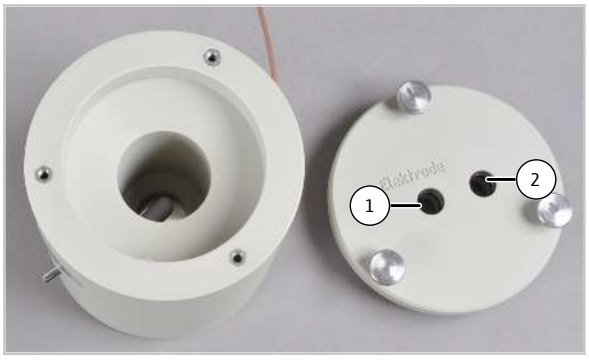


Fig. 26 Measuring cell "sensitive" with lid

1 Opening for combined electrode
2 Opening for direct injection and exhaust connection

Fig. 27 Equipped measuring cell

1 Combined electrode
2 Olive for connecting to the exhaust

The combined electrode is used for the "sensitive" and "high concentration" measuring cells. It combines the indicator electrodes (Ag), the generator cathode (Pt) and the gas inlet. The measuring gas hose can be connected directly to the electrode.

The combined electrode is rinsed thoroughly after measurement and stored in a dry place.

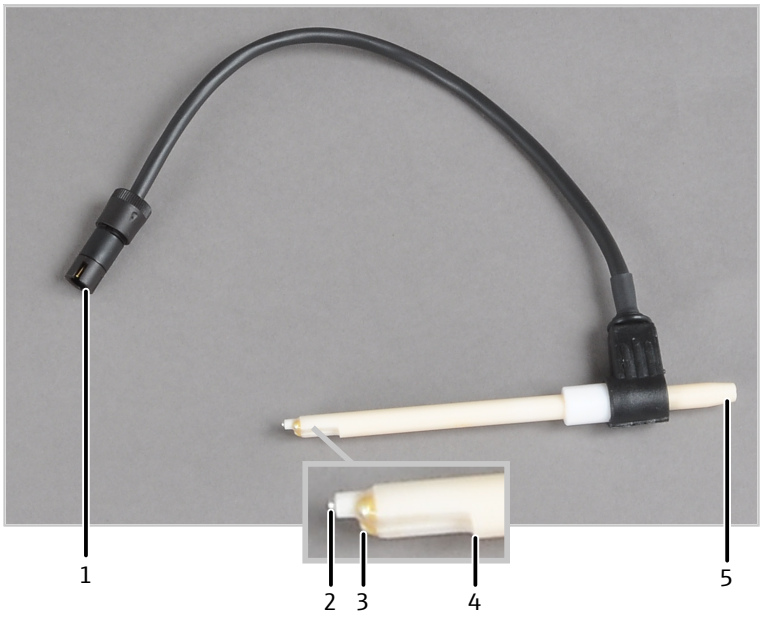


Fig. 28 Combined electrode

1 Connector plug
2 Generator cathode (Pt)
3 Indicator electrodes (Ag)
4 Gas inlet to measuring cell
5 Measuring gas connection (hose 20)

The "high concentration" measuring cell

The measuring cell has the same function as the "sensitive" measuring cell, but has a higher electrolyte volume. It is suitable for a chlorine content of 10 to 1000 µg, and is recommended in particular for TX determination of highly contaminated waste and of polymer samples with high PVC content.

Here, as well, the maintenance-free combined electrode is used.

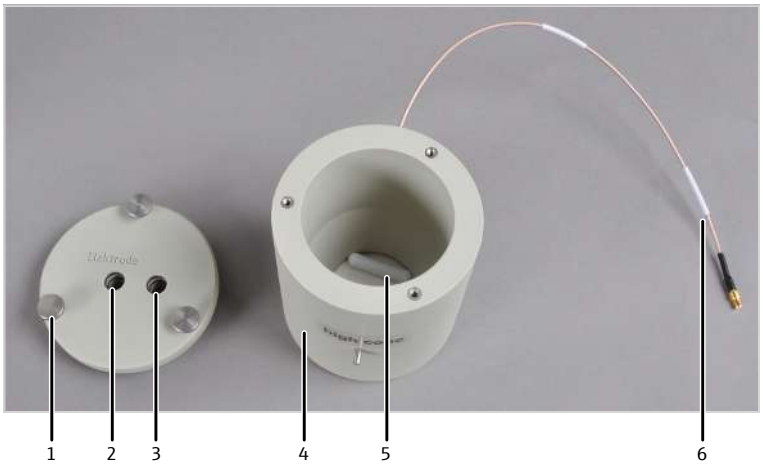


Fig. 29 Measuring cell "high concentration" with lid

1 Knurled head screw
2 Opening for combined electrode (labeled)
3 Opening for direct injection and exhaust connection
4 Electrode space with silver anode
5 Magnetic stirring rod
6 Electrical connection of the measuring cell

"high sensitive" measuring cell

This measuring cell is used for very low chlorine content (0.01 to 10 µg). The measuring cell is especially recommended for trace and ultra trace analysis in industrial quality control and for EOX determination.

The measuring cell consists of the base element with magnetic stirring rod and lid. The positions of the required components are marked inside the lid of the measuring cell.

The central borehole is intended for an adapter that is connected to the exhaust hose. It is used as an exhaust for the acetic acid vapors.

The small unmarked opening is used for direct injection into the cell. This opening is closed with a plug when measuring gas is introduced into the measuring cell from the combustion furnace.

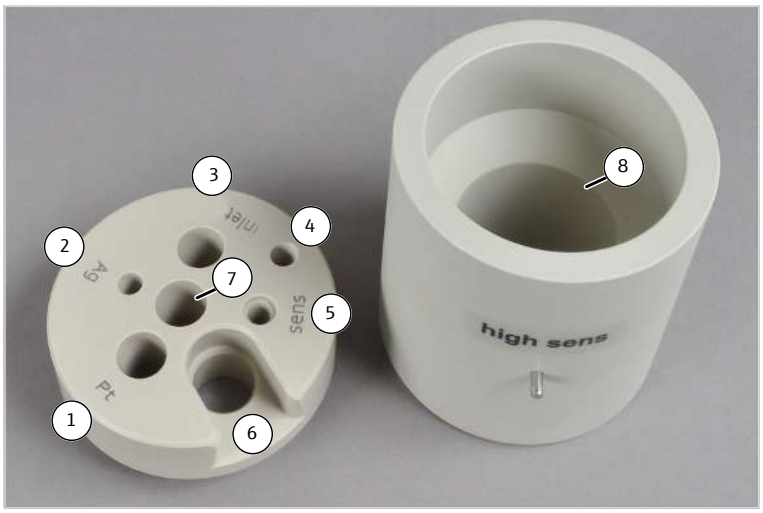


Fig. 30 "high sensitive" measuring cell with lid

1 Opening for the platinum electrode
2 Opening for the silver electrode
3 Opening for the gas inlet tube
4 Opening for direct injection
5 Opening for the sensor electrode
6 Opening for the reference electrode
7 Opening for exhaust connection
8 Measuring cell base element with magnetic stirrer rod

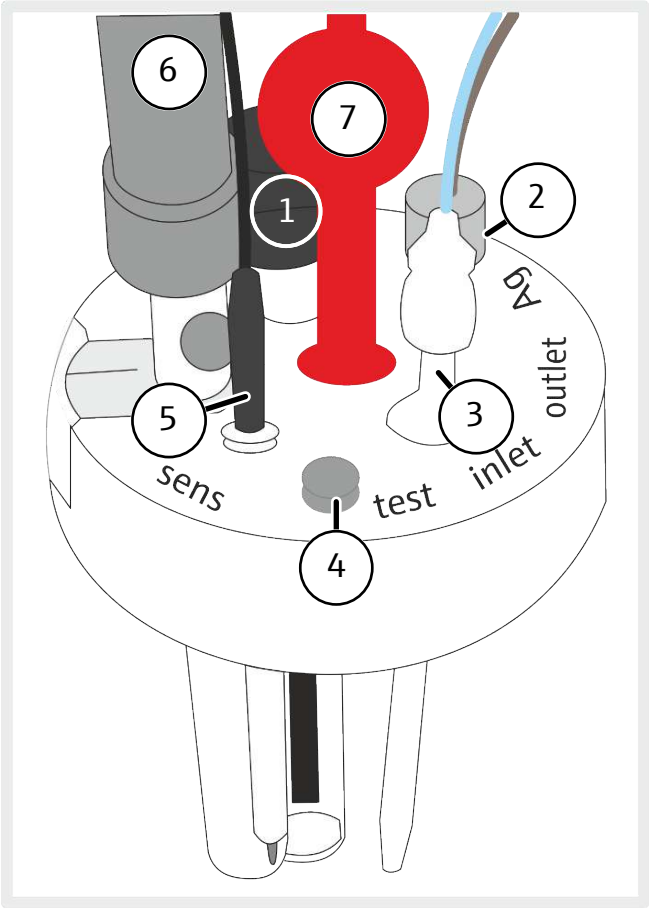


Fig. 31 Lid of the "high sensitive" measuring cell equipped with all electrodes

1 Platinum electrode
2 Silver electrode
3 Gas inlet tube with Teflon screw connection (for measuring gas)
4 Opening for direct injection, with plug
5 Sensor electrode
6 Reference electrode
7 Adapter for exhaust connection

Sensor and reference electrodes

The sensor and reference electrode are always inserted into the measuring cell together.

The sensor electrode has a chlorinated sensor pin which is inserted into the measuring cell, and a gold contact. The sensor pin is touch-sensitive. Scratch protection is applied to it for storage.

The reference electrode is supplied dry, with no bridging electrolyte. The same acetic acid electrolyte solution used for the measuring cell is used as a bridging electrolyte. The bridging electrolyte is applied via the filling opening. The reference electrode is ready for measurement after a run-in period of approx. 4 h in the measuring cell.

The filling opening of the reference electrode must be open during operation. For short-term storage, close the filling opening and place the dry protective cap on the electrode.

The openings in the lid of the measuring cell are designed to align the sensor pin of the sensor electrode toward the diaphragm of the reference electrode.

A pre-amplifier is applied to the reference electrode. Its connection cables connect the reference electrode with the sensor electrode and with the connection on the detection module.

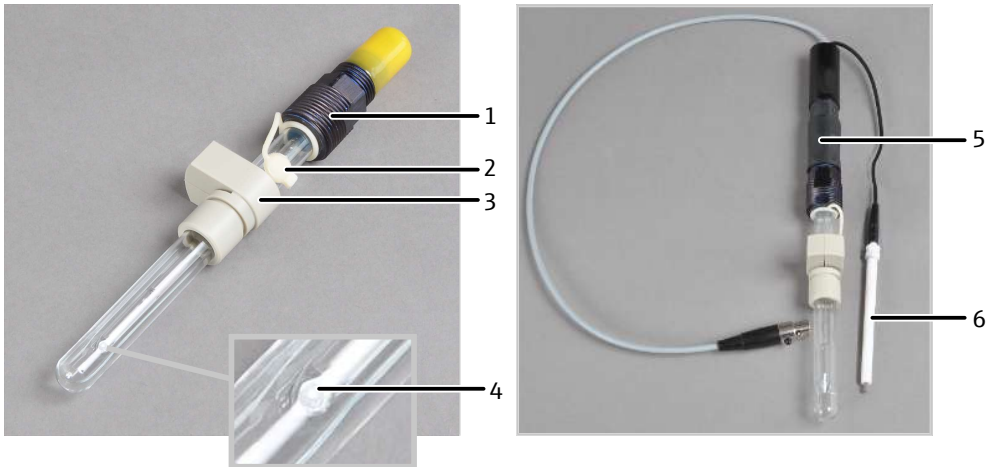


Fig. 32 Reference electrode and sensor electrode

1 Electrical connection
2 Filling opening for bridging electrolyte
3 Holder for the reference electrode in the measuring cell lid
4 Diaphragm
5 Pre-amplifier
6 Sensor electrode

A short hose with a T piece can be found in the accessories of the measuring cell. The hose and the T piece are applied to the adapter and connected to the exhaust hose. One side of the T piece remains open. The acetic acid vapors can be drawn out of the detection module effectively without the electrolyte solution evaporating too rapidly.

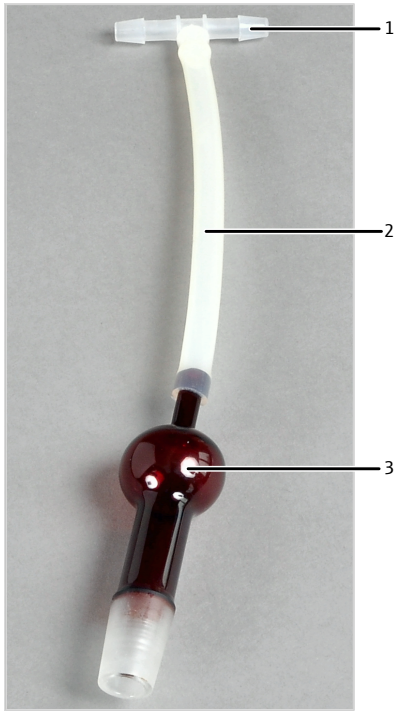


Fig. 33 Exhaust connection

1 T piece
2 Short hose
3 Adapter

Generator electrodes

A pair of generator electrodes generate the silver ions needed for chemical precipitation. The pair of electrodes consist of a platinum cathode with a salt bridge and a silver anode.

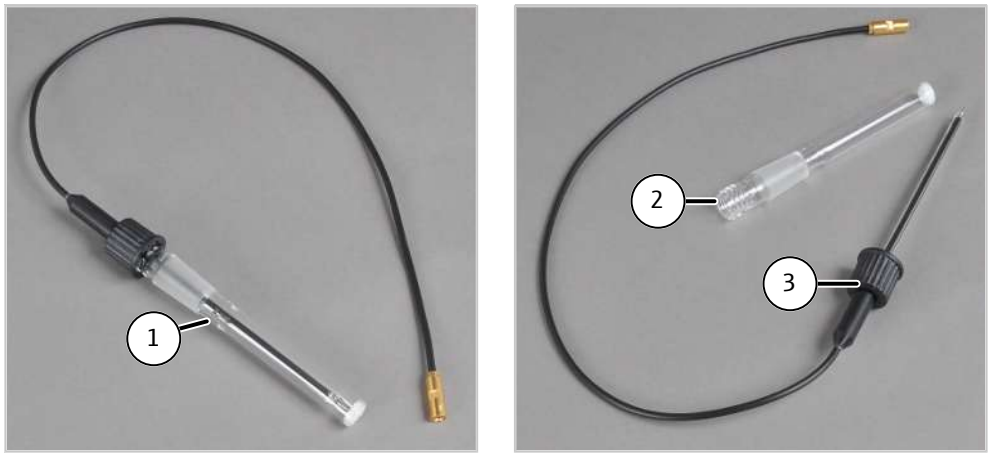


Fig. 34 Platinum electrode with salt bridge

1 Platinum electrode with salt bridge, complete
2 Salt bridge
3 Platinum electrode with screw-on cap and sealing ring

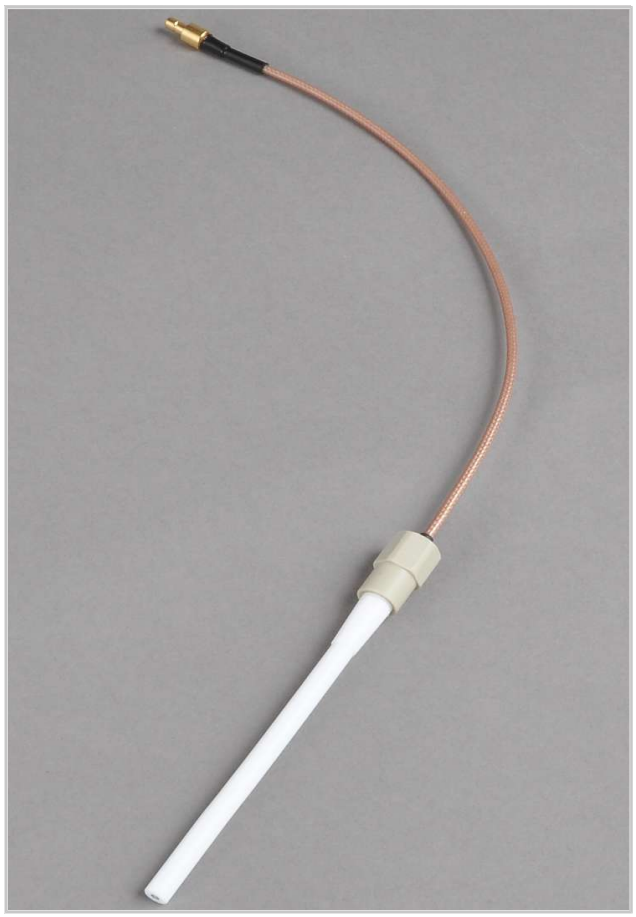


Fig. 35 Silver electrode

8.1.3 Connection

An LED is installed on the front of the detection module. The LED lights up when the module is switched on.

The power switch, the device fuse and the power connection are located on the rear of the module. The RS 232 interface for connection to the basic module ("Cl-Coul" interface) is also located on the rear of the module. The gas outlet for connection to the exhaust hose of the laboratory exhaust system is located at the bottom left of the rear panel.

Fig. 36 Rear of the chlorine detector

1 Interface to the basic module
2 Gas outlet
3 Power connection
4 Fuse holder
5 Equipment switch

The electrical connections for the measuring cells and electrodes are located on the inside of the detection module's rear panel. They cannot be interchanged. Each connector only fits into one terminal.

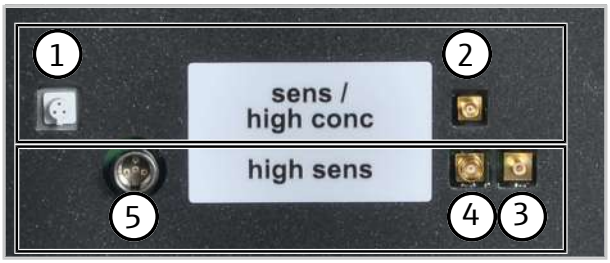


Fig. 37 Measuring cell connections

1 Combined electrode connection
2 Measuring cells "sensitive" and "high concentration" connection
3 Platinum electrode connection
4 Silver electrode connection
5 Sensor and reference electrode connection (via pre-amplifier)

Measuring gas transfer

A heated gas transfer line is used to transfer the measuring gas. It connects the combustion system of the basic module with the sulfuric acid container in the detection module.

The hose of the gas transfer line is connected to the sulfuric acid container connection with a banjo bolt with a conical nipple. The other end of the gas transfer line is connected to the auto-protection valve assembly in the basic device.

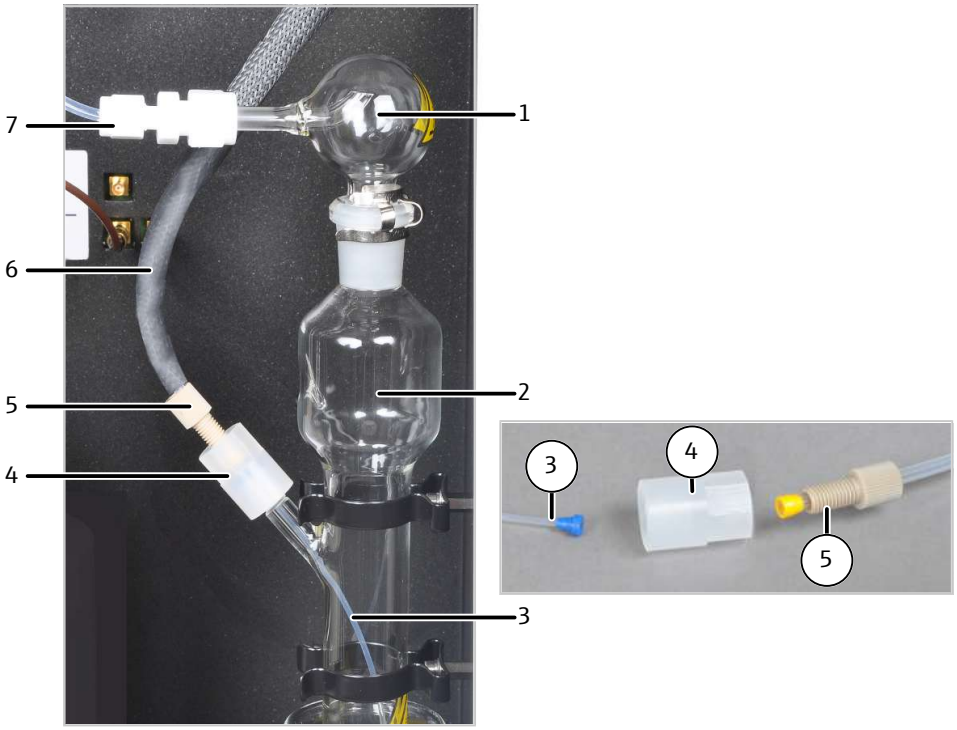


Fig. 38 Connection of the heated gas transfer lines to the sulfuric acid container

1 Safety attachment
2 Sulfuric acid container
3 Hose for measuring gas introduction
4 Connector
5 Banjo bolt
6 Heated gas transfer line
7 Measuring gas transfer to the measuring cell with PTFE connector

The measuring gas is introduced into the measuring cell via the safety attachment of the sulfuric acid container.

- The measuring gas hose is connected directly to the combined electrode with the "sensitive" and "high concentration" measuring cells.
- A cut glass tube is used to feed gas into the "high sensitive" measuring cell.

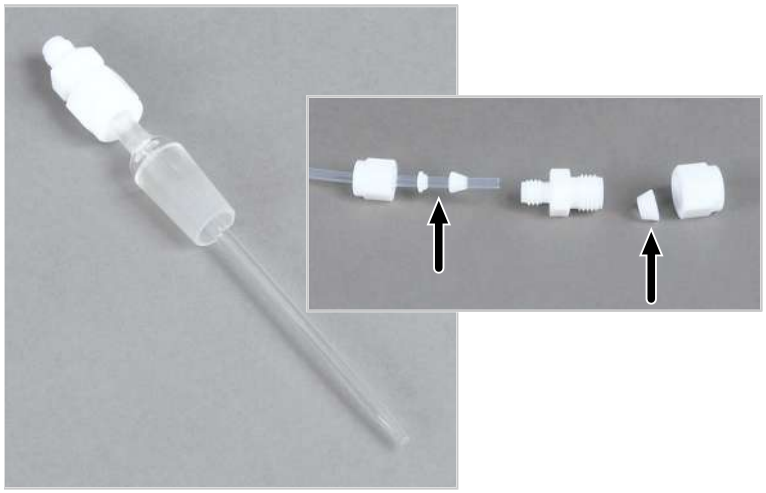


Fig. 39 Gas introduction tube with PTFE connector

In both cases, the measuring gas hose is fastened with the aid of a PTFE connector. Ensure the proper fit of the sealing rings!

Fig. 40 Gas introduction into the "sensitive" measuring cell

8.2 Installation

CAUTION

Risk of injury

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

- Handle glass parts with extreme caution.

NOTICE

Connecting or disconnecting electrical contacts may damage the sensitive electronic components of the basic module and of the detection module.

- Always connect the modules to power when they are switched off.

- Place the detection module to the immediate left of the basic module.
- Connect the supplied power cable to the power cable connection on the rear of the module and to an earthed socket. Observe the permissible maximum voltage!
- Connect the detection module with the basic module via the interface:
  CL-Coul interface on the rear of the detection module
  CL-Coul interface on the rear of the basic module
- Connect the "waste" outlet with the exhaust hose. Connect the hose with the exhaust system, or route the hose into a vent.

Installing the detection module

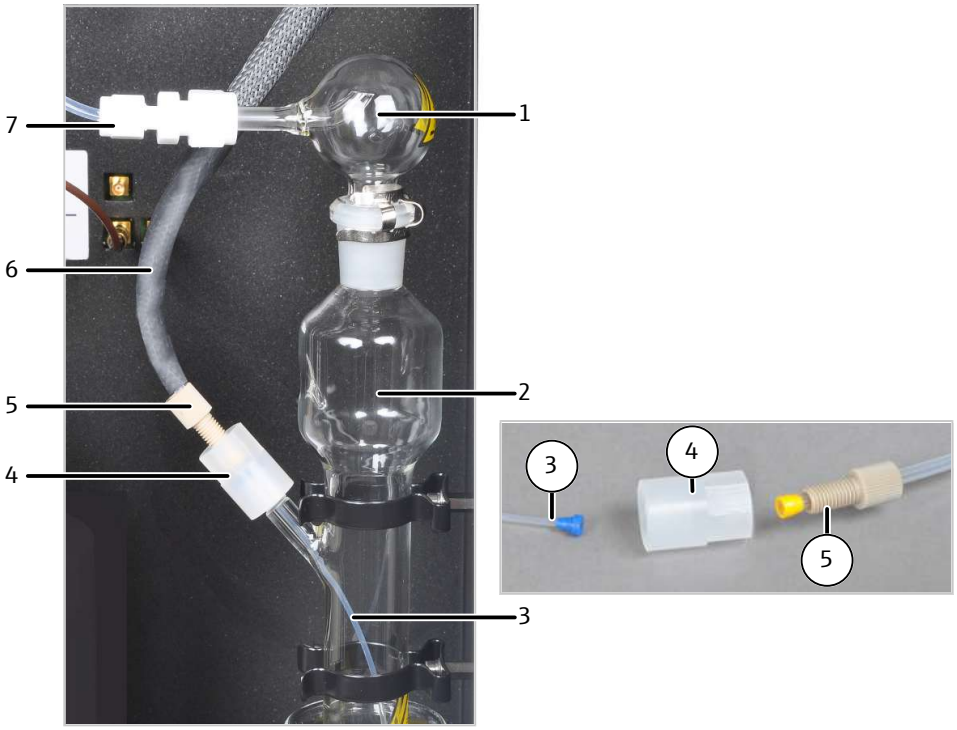


Fig. 41 Connection of the heated gas transfer lines to the sulfuric acid container

1 Safety attachment
2 Sulfuric acid container
3 Hose for measuring gas introduction
4 Connector
5 Banjo bolt
6 Heated gas transfer line
7 Measuring gas transfer to the measuring cell with PTFE connector

- Insert the sulfuric acid container in the holders.
- Route the gas transfer line through the top right opening of the detection module.
- Connect the gas transfer line to the sulfuric acid container:
  - Insert the thin hose (3) into the sulfuric acid container.
  - Connect the connector (4) to the sulfuric acid container.
  - Connect the gas transfer line via the banjo bolt (5) on the connector.
    NOTICE! Ensure the proper fit of the conical nipples!
- Place the safety attachment on the sulfuric acid container and attach the safety clip.
- Connect hose 20 with the safety attachment. Connect the hose to the measuring cell later.
- Connect the gas transfer line in the basic module:
  - Route the free end of the gas transfer line through the top left opening on the basic module.
  - Connect the gas transfer line to the auto-protection valve assembly.
  - Connect the heating cable of the gas transfer line to its socket.

Fig. 42 Connection of the gas transfer line in the basic module

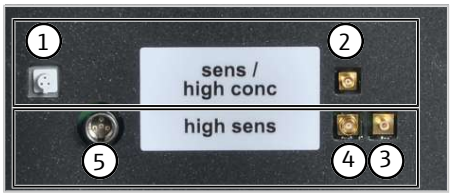
Left Connection to the auto-protection valve assembly
Right Heating cable connection

Connection of the detection module is complete.

Inserting measuring cells

The "sensitive" and "high concentration" measuring cells are inserted into the detection module as follows:

- Insert the measuring cell with magnetic stirring rod and lid into the detection module.
- Fill the measuring cell with electrolyte solution.
- Insert the combined electrode into the marked opening of the measuring cell.
- Fasten hose 20 from the sulfuric acid container to the connection of the combined electrode with the PTFE connector (1).
- Connect the olive (2) with the exhaust hose (hose 21) in the detection module.



- Connect the combined electrode and the measuring cell to the inside of the rear panel of the detection module:
  Combined electrode connection (1)
  Measuring cell connection (2)
  Do not use connections (3) to (5).

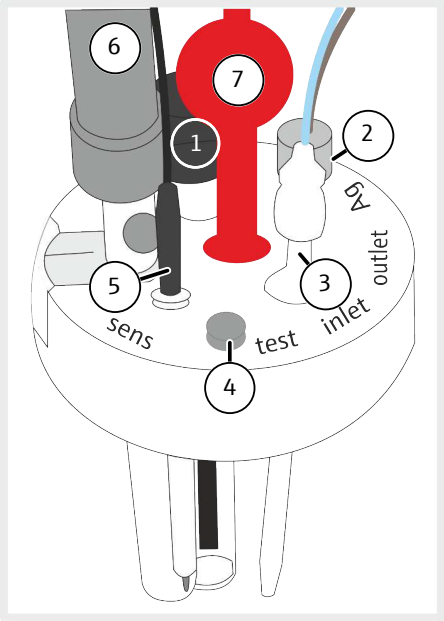
The "high sensitive" measuring cell is inserted into the detection module as follows:

NOTICE

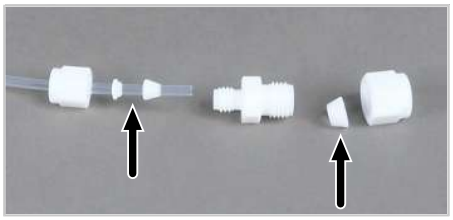
Risk of damage to the sensor electrode

The sensor pin and the gold contact of the sensor electrodes are sensitive to touch.

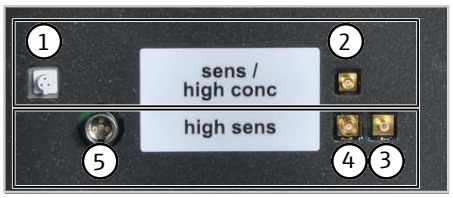
- Apply scratch protection to the sensor pin for storage.
- Rinse the sensor pin with ultrapure water before use or to clean it. Do not touch it again after this. Do not dry the pin or wipe it dry!
- Before use or to clean it, wipe the gold contact with a cloth and some ethanol. Do not touch it again after this.



- Insert the measuring cell with magnetic stirring rod and lid into the detection module.
- Fill the measuring cell with electrolyte solution.
- Insert the components in the following openings:
  Platinum electrode with salt bridge (1): "Pt" opening
  Silver electrode (2): "Ag" opening
  Gas inlet hose (1): "Inlet" opening
  Sensor electrode (5): "Sens" opening
  Reference electrode (6): large "ref" opening
  Adapter (7): Center "outlet" opening
  The openings on the lid align the sensor electrode and the reference electrode toward each other.
- Close the opening for direct injection (4, "test") with a plug.
- Connect the short hose with the T piece to the adapter (7). Connect the exhaust hose in the detection module (hose 21) with a limb of the T piece.
- Place the pre-amplifier on the reference electrode. Connect the pre-amplifier with the sensor electrode.



- Connect hose 20 from the sulfuric acid container to the gas inlet tube via the PTFE connector.
  NOTICE! The conical nipples of the PTFE connector must be in the proper position on the hose. Otherwise, gas leaks may ensue.



- Connect the electrode on the inside of the rear panel of the detection module:
  Platinum electrode connection (3)
  Silver electrode connection (4)
  Sensor and reference electrode connection (5)
  Do not use connections (1) and (2).

8.3 Operation

8.3.1 Preparing the measuring cell

Preparing the measuring cell includes the following steps:
- Preparing the electrolyte solution
- Performing the end point routine

8.3.1.1 The "sensitive" and "high concentration" measuring cells

The measuring cells are similar in function. The "high concentration" measuring cell works with a larger electrolyte solution volume.

Preparation of the electrolyte solution

WARNING

Risk of chemical burns

100 % acetic acid (glacial acetic acid), concentrated nitric acid and thymol can cause severe chemical burns. Methanol is a toxic, highly flammable material.

- Wear protective clothing when preparing the electrolyte solution. Work under an extractor.
- Observe all instructions and specifications in the safety data sheets.

⇨ Reagents required: 200 ml acetic acid 100 % (glacial acetic acid), 4 ml concentrated nitric acid, 4 g gelatin, 1.0 g thymol, 0.3 g thymol blue, 500 ml methanol

▸ Solution A:
Fill 500 ml of water into a 1000-ml volumetric flask, add 4 ml of HNO_3 (conc.), carefully add 200 ml of acidic acid and top up with water to the marking.

▸ Solution B1:
Mix 4 g of gelatin in a beaker with 400 ml of water, allow to swell for 3 hours and then dissolve whilst heating to 35 to 45 °C.
The excess gelatin will sediment at the bottom of the beaker. Only use the clear supernatant. Filter the solution, if necessary.

▸ Solution B2:
Dissolve 1.0 g of thymol and 0.3 g of thymol blue in a beaker with 500 ml of methanol.

▸ Solution B:
After solution B1 has cooled down to 18 to 22 °C, slowly add it to solution B2 while stirring, transfer into a 1000-ml volumetric flask and top up with water to the marking.

▸ Solution C, finished electrolyte:
Pipette 8 ml of solution B in a 100 ml measuring cylinder and fill with solution A to 100 ml, **or**
pipette 40 ml of solution B in a 500 ml measuring cylinder and fill with solution A to 500 ml.

✓ The electrolyte solution is finished.

Storage and shelf life of the electrolyte solutions:

- When stored at 4 ±3 °C in well-sealed bottles, solutions A and B can be kept for approx. 6 months.
- The finished electrolyte solution (solution C) can be kept for approx. 30 days if kept in a well-sealed glass container at 20 to 25 °C.

Performing the end point routine

An end point routine is necessary after every electrolyte change. The end point routine is used to adjust the electrolyte to the optimal operating range of the measuring cell. The operating point of the measuring cell is: 1500 to 5000 Counts .

- Start the end point routine via the **System** | **End point routine** menu item.
- Remove the olive from the lid of the measuring cell. Dose the HCl solution directly into the measuring cell when instructed to by the software.
  "sensitive" measuring cell: 200 µl of 0.01 N HCl
  "high concentration" measuring cell: 200 µl of 0.1 N HCl
- Directly after dosing, activate the end point routine by clicking on **[OK]**.
- The **End point routine** status is displayed in the **Status analyzer** window during this routine. After the end point routine, the **Stand-by titration** and the current indicator value is displayed.
  ✓ The system is ready for measurements.

The determined operation point of the combined electrode is displayed in the **System** | **Component test** menu item in the **Chlorine** tab.

Protecting the combined electrode

Observe the following to protect the combined electrode against unnecessary wear:
- Always add fresh electrolyte to the measuring cell before the end point routine.
- Do not carry out the end point routine several times in a row.

8.3.1.2 The "high sensitive" measuring cell

Preparation of the electrolyte solution

Measurements with the "high sensitive" measuring cell require an electrolyte solution. The electrolyte solution is also used as a bridge electrolyte for the reference electrode.

WARNING

Risk of chemical burns

100 % acetic acid (glacial acetic acid) can cause severe chemical burns. Gases may develop during shaking.

- Wear protective clothing when preparing the electrolyte solution. Work under an extractor.
- Observe all instructions and specifications in the safety data sheet.

⇨ Reagents required: 800 ml acetic acid 100 % (glacial acetic acid), 2.7 g sodium acetate p.a. (CH_3COONa), anhydrous

- Dilute 2.7 g of sodium acetate in 200 ml of ultrapure water in a 1-liter volumetric flask.
- Carefully add 800 ml of glacial acetic acid. Keep moving the flask while pouring the acid. Carefully shake the mixture.
  NOTICE! Do not exceed the specified amounts of water and glacial acetic acid. Do not fill the flask up to the 1-liter mark (volume contraction).

Performing the end point routine

The end point routine is used to set the electrolyte to the optimum operating point of the sensor electrode within the operating range of the titration cell.

- Operating range: 1000 to 10000 Counts
- Optimal operation range: 3000 Counts

Automatic end point routine

As soon as the indicator value is outside the operating range of the titration cell, the system automatically triggers an end point routine. This routine may also be triggered between two measurements of a multiple determination. The End point routine status is displayed in the Status analyzer window during this routine.

Indicator value is higher than the operating range:

- Silver ions are automatically produced. The electrolyte is set to the optimum operating range of 3000 Counts.

Indicator value is lower than the operating range:

- The software prompts the user to add the following solution to the measuring cell: 100 µl hydrochloric acid (HCl, 10 mg/l HCl)
- If the indicator value rises above the operating range again, silver ions will be produced automatically. The electrolyte is set to the optimum operating range.

When working with a multi matrix sampler, the addition of chloride ions to the measuring cells can be performed automatically if the indicator value goes below the operating range while an analysis sequence is in progress. For this, the user must have prepared a suitable organic chlorine solution (10 to 100 mg/l) and placed it in the intended position (110) on the sample rack.

After the end point routine, the measuring cell requires approx. 15 min to achieve a stable cell potential. During this time, a negative drift can occur with indicator values below 3000 Counts.

Manual end point routine

For indicator values within the operating range, the end point routine can be started manually via the **System** | **End point routine** menu item.

After the end point routine, the current indicator value is displayed in the **Status analyzer** window. If the current indicator value is within the operating range and the drift is stable, the system is ready for measurements

8.3.2 Operating the analysis system

- ▶ Place the measuring cell with electrodes and electrolyte solution in the detection module and connect it to electricity.
- ▶ Switch on the basic module and the detection module.
  - ✓ The devices boot up. The status LED on the front of the basic module light up in green after approx. 30 s.
  - ✓ The LED on the front of the detection module flashes during the run-in period. Depending on the detector, the run-in period can take up to 30 min. After this, the LED will light up continuously. Starting a measurement is only then possible.
- ▶ Open the gas supply and set the required gas pressure.
- ▶ Switch on the PC.
- ▶ Start the control and analysis software and login with your username and password.
- ▶ Initialize the analysis system by clicking on **[Initialize analyzer]**.
  - ✓ The initialization and automatic detection of all connected components will be carried out.
- ▶ Ready the samples.
- ▶ Activate a pre-existing method via the **Method** | **Method - activate** menu item.
- ▶ Alternatively: Create a new method in the **Method** | **Method - new** menu. Select the measurement parameter in the method. Release and activate the method.

- Select **Start** | **Start - Analysis** in the menu.
- Select an analysis group or create a new one and confirm via **[OK]**.
- Create an analysis sequence.
- Enter the sample ID for all sample in the **Name** field.
- Release all sequence lines
- Confirm the entries with **[OK]**.
- Click the **[Start Measurement]** button.
  - ✓ The prepared analysis sequence is processed.

For manual sampling, follow the instructions in the software.

8.3.3 Notes for measuring operations

CAUTION

Risk of respiratory problems due to leaking acetic acid vapors

The electrolyte solution of the "high sensitive" measuring cells contains high concentrations of acetic acid.

- Ensure that the exhaust hose is connected to the measuring cell.
  Check if the exhaust hose is connected to the "waste" outlet on the rear of the detection module, and that it is connected to the exhaust system.
- Before measurement operations, always close the front door of the detection module and switch on the laboratory exhaust system.

WARNING

Risk of chemical burns

Concentrated sulfuric acid is used in the detection module as a drying agent. The concentrated acid can lead to severe chemical burns.

The 100 % acetic acid (glacial acetic acid), nitric acid and thymol used to create the electrolyte solution can lead to severe chemical burns.

- Wear protective clothing when working with these hazardous substances.
- Observe all instructions and specifications in the safety data sheets.

- Fill the sulfuric acid container with fresh concentrated sulfuric acid every day (→ "Replacing the sulfuric acid and cleaning the sulfuric acid container." 145).

The "sensitive" and "high concentration" measuring cells

Fill the measuring cells with fresh electrolyte solution daily:
- "sensitive" measuring cell: 15 to 20 ml
- "high concentration" measuring cell: 120 ml

"high sensitive" measuring cell

Fill-up the measuring cell daily with electrolyte solution: to approx. 65 ml

Change the electrolyte solution:
- Once per week
- If analytical problems occur
- If crystalline deposits form

Open the refill opening of the reference electrode during operation.

9 Sulfur analysis with the S module 5100 (basic. MPO)

9.1 Function and design

9.1.1 Function and measuring principle

Expansion of the basic module with the detection module allows determination of the sulfur content in solid, liquid, paste-like, viscous and gaseous samples via UV flourescence.

The measuring gas is created during incineration of organic sulfur compounds in the basic module. It contains sulfur dioxide (SO_2).

$R\text{-}S + O_2 \rightarrow SO_2 + CO_2 + H_2O$

R: Hydrocarbon residue

The detection is performed using the UV fluorescence method. Sulfur dioxide (SO_2) excited via UV light emits a characteristic flourescence (220 to 420 nm). This flourescence is measured. The SO_2 concentration is determined from the changes to the flourescent intensity.

9.1.2 Design

The detection module allows determinations of the sulfur content via UV flourescence. All components required for determination are mounted inside the sealed housing.

Fig. 43 Basic module with detection module and sampling module

This analysis gas containing SO_2 is excited to flourescence via the radiation of a UV lamp. The intensity of the fluorescence is determined with a photomultiplier (PMT).

To determine the sulfur content without interference from simultaneous increased nitrogen content the patented MPO (microplasma optimization) technology has been developed. The MPO technology removes the interfering nitrogen monoxide from the measuring gas. This important, for example, during analysis of diesel fuels containing cetane improvers.

The detection module is available with and without the MPO option. The MPO option is not suited for multi-element methods that determine multiple elements simultaneously. It can be switched on and off as needed via the control and analysis software.

Working with a method with the active MPO requires calibration performed with the MPO active. Otherwise the measurement results will be too low. Conversely, use of calibration with an active MPO for a method with no MPO can lead to false measurement results that are too high.

9.1.3 Connection

The equipment door at the front is closed tightly and cannot be opened. An LED is installed on the door. The LED flashes during the run-in period of the detection module and lights up permanently when the module is operational.

The device switch for switching the module on and off is located on the top right of the rear of the device (when viewed from the front). The device fuse and power connection are located beneath it.

Communication with the basic module is performed via a 9 pin interface cable. The interface is labeled "S-UVF".

The hose for the measuring gas coming from the basic module is connected to the "sample in" gas inlet. The gas outlet is labeled "sample out".

The "Service" interface and the programming button are only required for service purposes.

Fig. 44 Rear of the sulfur detector

1 Chemical ozone decomposer (MPO)
2 Measuring gas inlet
3 Measuring gas outlet
4 Service interface and programming button.
5 Interface to the basic module
6 Power connection
7 Fuse holder
8 Equipment switch

The diagram on the rear explains the connector allocation.

9.2 Installation

NOTICE

Connecting or disconnecting electrical contacts may damage the sensitive electronic components of the basic module and of the detection module.

- Always connect the modules to power when they are switched off.

- Place the detection module to the left of the basic module. In the event of a series of detection modules: place the detection module to the left or right of the others.
- Connect the supplied power cable to the power cable connection on the rear of the module and to an earthed socket. Observe the permissible maximum voltage!
- Connect the detection module with the basic module via the interface:
  S-UVF interface on the rear of the detection module
  S-UVF interface on the rear of the basic module
- Connect the measuring gas hose of the basic module to the "sample in" gas inlet on the rear of the module.
- Leave the "sample out" outlet unconnected or connect it to the measuring gas inlet of the next detection module.
- For detection modules with MPO technology: Install the chemical ozone decomposer on the rear of the module:
  - Fasten the two holding clamps with the supplied screws.
  - First press the chemical ozone decomposer into the upper holding clamp and then into the lower one.
  - Connect the hose from the "waste (MPO)" outlet to the ozone decomposer. Do not pull the hose out of the device!

  ✓ Connection of the detection module is complete.

Fig. 45 Chemical ozone decomposer

9.3 Operation

CAUTION

Risk of respiratory problems due to leaking ozone

If the gas hoses have not been properly connected to the ozone generator, ozone may leak out of the detection module.

- If you can smell ozone, switch off the device and check the connection of the gas hoses on the ozone generator.

- Switch on the basic module and the detection module.
  - ✓ The devices boot up. The status LED on the front of the basic module light up in green after approx. 30 s.
  - ✓ The LED on the front of the detection module flashes during the run-in period. Depending on the detector, the run-in period can take up to 30 min. After this, the LED will light up continuously. Starting a measurement is only then possible.
- Open the gas supply and set the required gas pressure.
- Switch on the PC.
- Start the control and analysis software and login with your username and password.
- Initialize the analysis system by clicking on **[Initialize analyzer]**.
  - ✓ The initialization and automatic detection of all connected components will be carried out.
- Ready the samples.
- Activate a pre-existing method via the **Method** | **Method - activate** menu item.
- Alternatively: Create a new method in the **Method** | **Method - new** menu. Select the measurement parameter in the method. Release and activate the method.
- Select **Start** | **Start - Analysis** in the menu.
- Select an analysis group or create a new one and confirm via **[OK]**.
- Create an analysis sequence.
- Enter the sample ID for all sample in the **Name** field.
- Release all sequence lines
- Confirm the entries with **[OK]**.
- Click the **[Start Measurement]** button.
  - ✓ The prepared analysis sequence is processed.

For manual sampling, follow the instructions in the software.

10 Sulfur analysis with the S module 5100 coulometric

10.1 Function and design

10.1.1 Function and measuring principle

Expansion of the basic module with the detection module allows the determination of the sulfur content in solids, liquids, and gases via microcoulometric titration.

The organic sulfur compounds are incinerated into a mixture of sulfur dioxide (SO_2) and sulfur trioxide (SO_3) in the basic module. Both oxides are created in a fixed relation. Carbon dioxide and water is also created during this incineration.

The amount of SO_2 is proportional to the total sulfur amount in the sample.

The measuring gas flow is first dried and then routed to the detection module via a transfer line. In the measuring cell, the sulfur oxides dissolve in the electrolyte and react with iodine. This decreases the cell potential.

$R\text{-}S + O_2 \rightarrow SO_2 + SO_3 + CO_2 + H_2O$

$2\ H_2O + SO_2 + I_2 \rightarrow H_2SO_4 + 2\ HI$

R: Hydrocarbon residue

After a specified accumulation time dependent of the sulfur content of the sample, titration begins. The iodine ions are oxidized back to iodine at the anode. This increases the cell potential. The end point of the iodometric titration has been reached when the measuring cell has again reached its original potential.

With titration and end point routine: The electrode reactions consist of an anode reaction (+) and a cathode reaction (-).

Anode (+): $2\ I^- \rightarrow I_2 + 2\ e^-$

Cathode (-): $2\ H^+ + 2\ e^- \rightarrow H_2$

10.1.2 Design

The detection module consists of the following main components:

- Measuring cell with electrodes
- NOx and HX absorbers for gas purification
- Gas inlet tube
- Magnetic mixer
- Interface to the basic module

A easily-opened door for changing the electrolyte solution is installed on the front of the detection module. For maintenance purposes, the door can be removed.

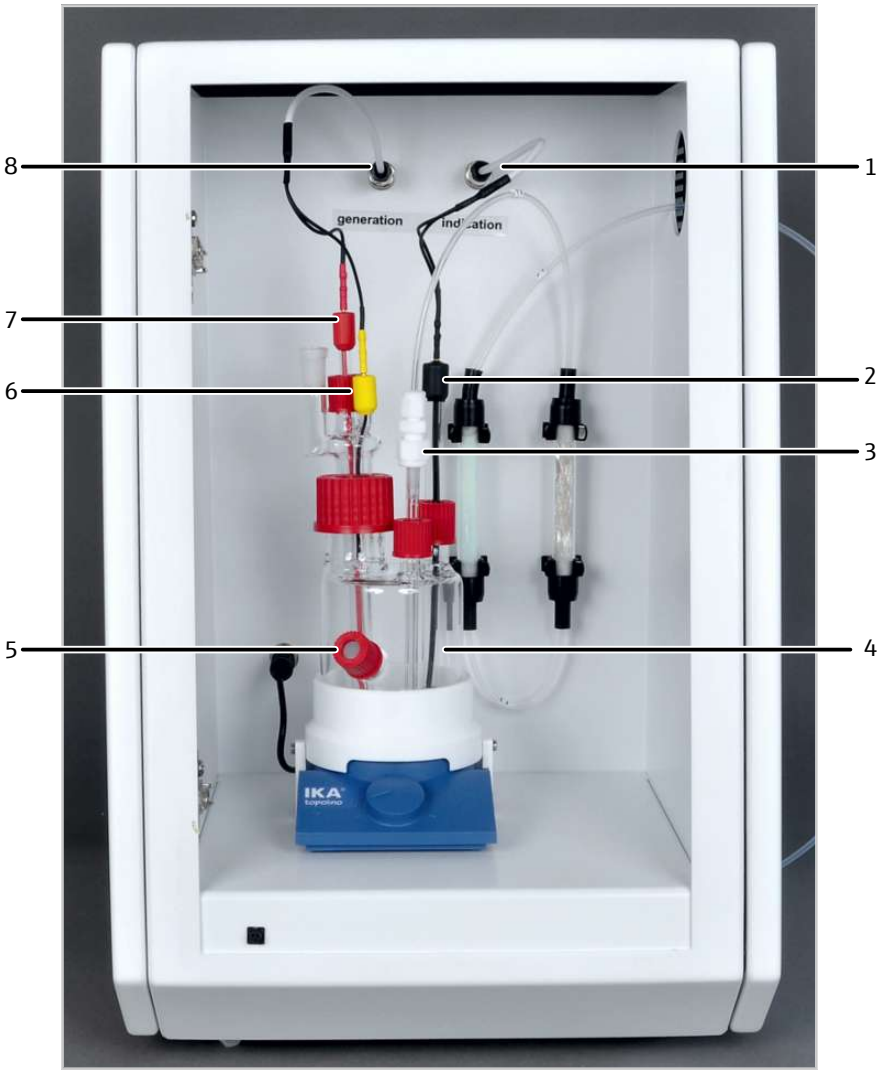


Fig. 46 Coulometric sulfur detector with measuring cell (without door)

1 Indicator electrodes connection
2 Indicator electrode (black)
3 Gas inlet
4 Measuring cell
5 Port for manual dosing
6 Anode (yellow)
7 Cathode (red)
8 Generator electrodes connection

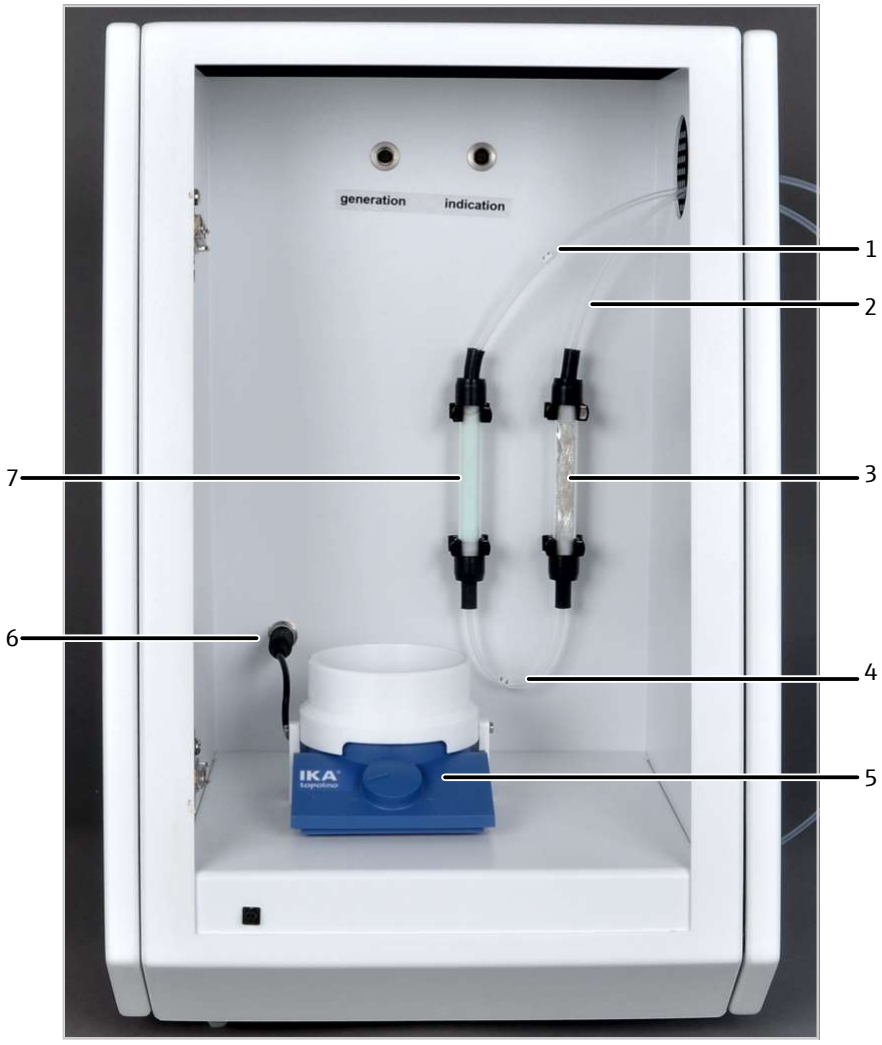


Fig. 47 Coulometric sulfur detectors without measuring cell

1 Measuring gas hose from the basic module (hose 71)
2 Measuring gas hose to the measuring cell (hose 72)
3 HX absorber
4 Hose 73
5 Magnetic mixer with controls
6 Magnetic mixer connection
7 NOx absorber

Measuring cell

The measuring cell is equipped with electrodes for generation and indication. The electrodes are color-coded:

- Generation: Yellow anode, red cathode
- Indication: Black indicator electrodes

The measuring gas is routed into the measuring cell via the gas inlet tube. The solution for the end point routine, the sodium sulfate solution (Na_2SO_3) and the solutions for the cell measurements are dosed, for example, via the port for manual dosing.

The measuring cell must be filled with approx. 100 ml electrolyte solution (approx. to the height of the manual dosing port).

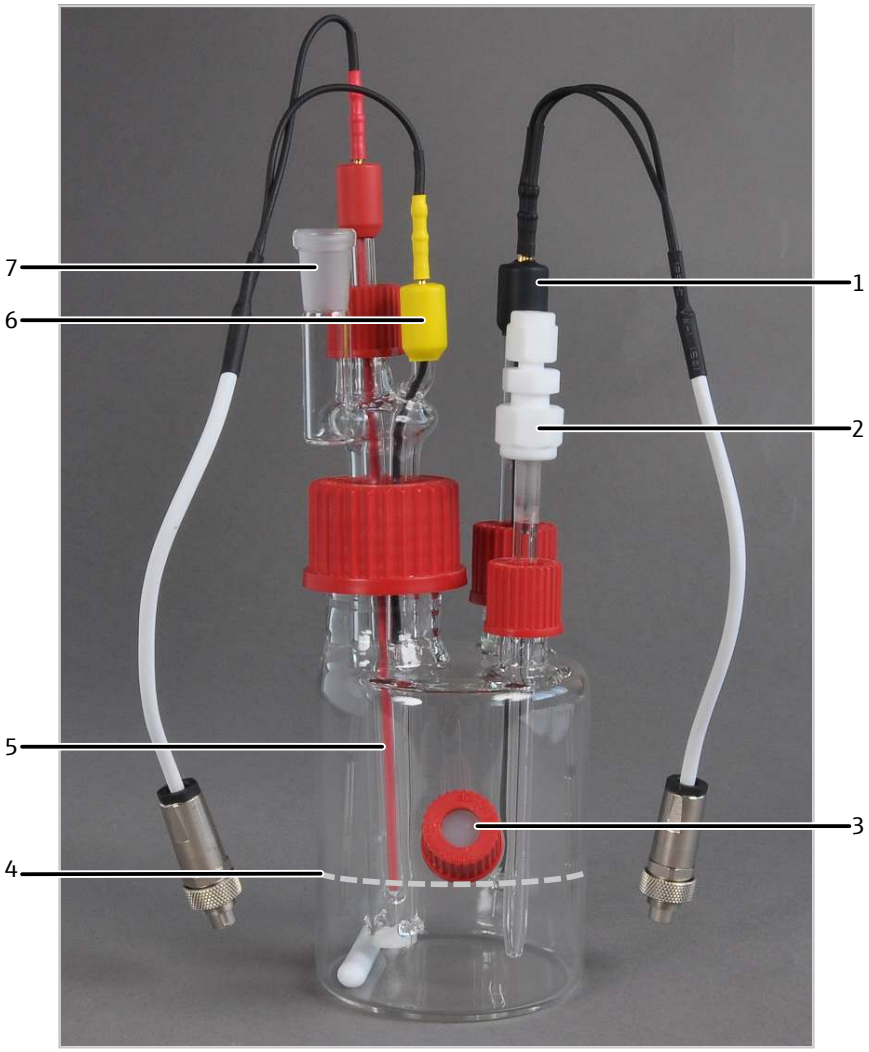


Fig. 48 Coulometric measuring cell

1 Indicator electrodes (black)
2 Gas inlet tube
3 Port for manual dosing
4 Electrolyte solution filling height
5 Cathode (red)
6 Anode (yellow)
7 Gas outlet

Electrodes

A diaphragm is located between the electrodes for generation (anode and cathode). The diaphragm is only permeable for sulfations. This means that the diaphragm prevent false analysis results due to undesired compounds.

The electrodes for indication are platinum electrodes.

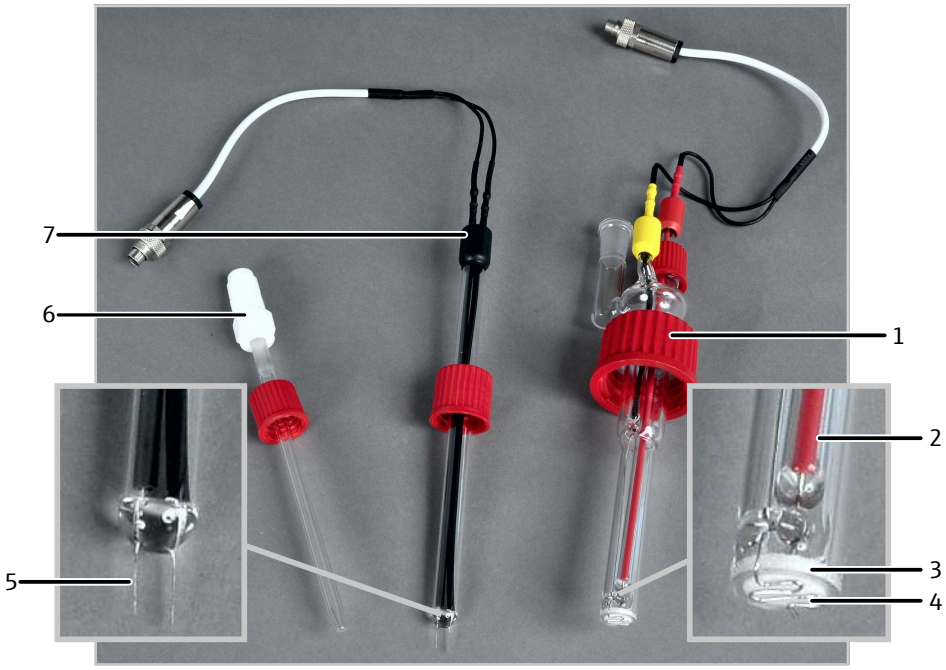


Fig. 49 Generator and indicator electrodes, gas inlet tube

1 Generator electrodes (with union nut)
2 Cathode (inside)
3 Diaphragm
4 Anode (outside)
5 Platinum electrodes
6 Gas inlet tube
7 Indicator electrodes

Absorber

To clean the measuring gas, two absorbers are installed in the detection module. The absorbers remove elements from the measuring gas that could disrupt the analysis.

The NOx absorber removes nitrogen oxides (NO_x) from the measuring gas. High nitrogen oxide content has an influence on the analysis and leads to false results. The filling of the absorber is normally green. If the color changes to yellow or light brown, the filling must be replaced.

The HX absorber removes hydrogen halides (HX with X = Cl, Br, I) from the measuring gas. Hydrogen halides disrupt the analysis due to cross-sensitivities. The absorber contains silver wool. The silver wool must be replaced if the color changes from metallic silver to dark gray.

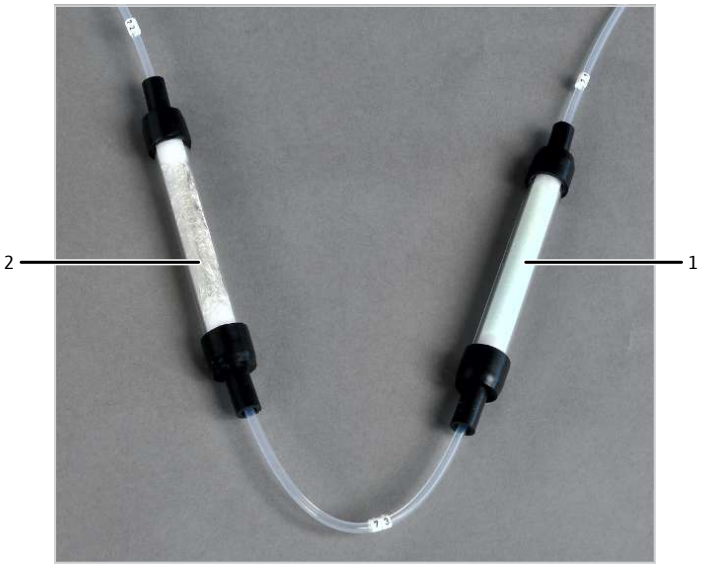


Fig. 50 NOx absorber and HX absorber

1 NOx absorber 2 HX absorber

Hose diagram

Labeled hoses connect the measuring cell with the other components in the detection module. The numbers in the hose diagram correspond to the labels on the hoses.

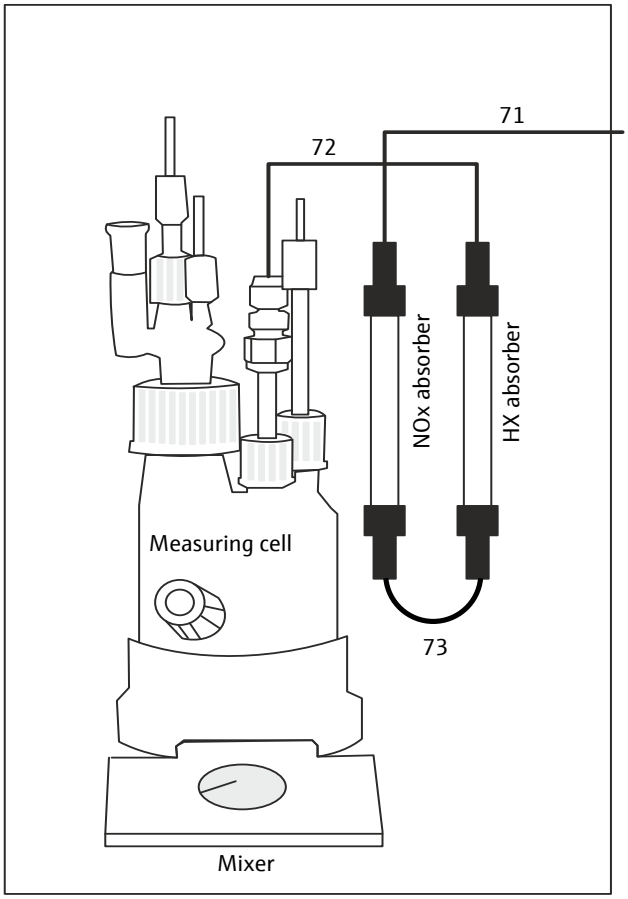


Fig. 51 Hose diagram

10.1.3 Connection

The electrical connection and the interface to the basic module can be found on the rear of the detection module.

The device switch for switching the detection module on and off is located on the top right of the rear of the device (when viewed from the front). The device fuse and power connection are located beneath it.

Communication with the basic module is performed via a 9 pin interface cable. The interface on the rear of the device is labeled "S-Coul".

Fig. 52 Rear of the coulometric sulfur detector

1 Power connection, fuse holder, device switch
2 Interface to the basic module

10.2 Installation

CAUTION

Risk of injury

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

- Handle glass parts with extreme caution.

NOTICE

Connecting or disconnecting electrical contacts may damage the sensitive electronic components of the basic module and of the detection module.

- Always connect the modules to power when they are switched off.

NOTICE

The detection module cannot be operated together with optical detectors.

- Do not connect the detection module together with an optical detector.

- Place the detection module to the left of the basic module.
- Connect the supplied power cable to the power cable connection on the rear of the module and to an earthed socket. Observe the permissible maximum voltage!
- Connect the detection module with the basic module via the interface:
  S-Coul interface on the rear of the detection module
  S-Coul interface on the rear of the basic module
- Route the measuring gas hose (hose 71) from the basic module through the side opening on the detection module. Connect the hose to the top of the NOx absorber.
- Connect the outlet of the HX absorber to hose 72.
  Hose 72 is connected to the measuring cell later.
- Check that the NOx absorber and the HX absorber are connected via hose 73.
- Insert the electrodes into the measuring cell as displayed in the illustration.
- Insert the gas inlet tube into the measuring cell. Connect the gas inlet tube with hose 72 via the PTFE connector.
- Connect the electrodes to the "Generation" and "Indication" connections.
  The connections cannot be mixed up.

  ✓ Connection of the coulometric sulfur detector is complete.

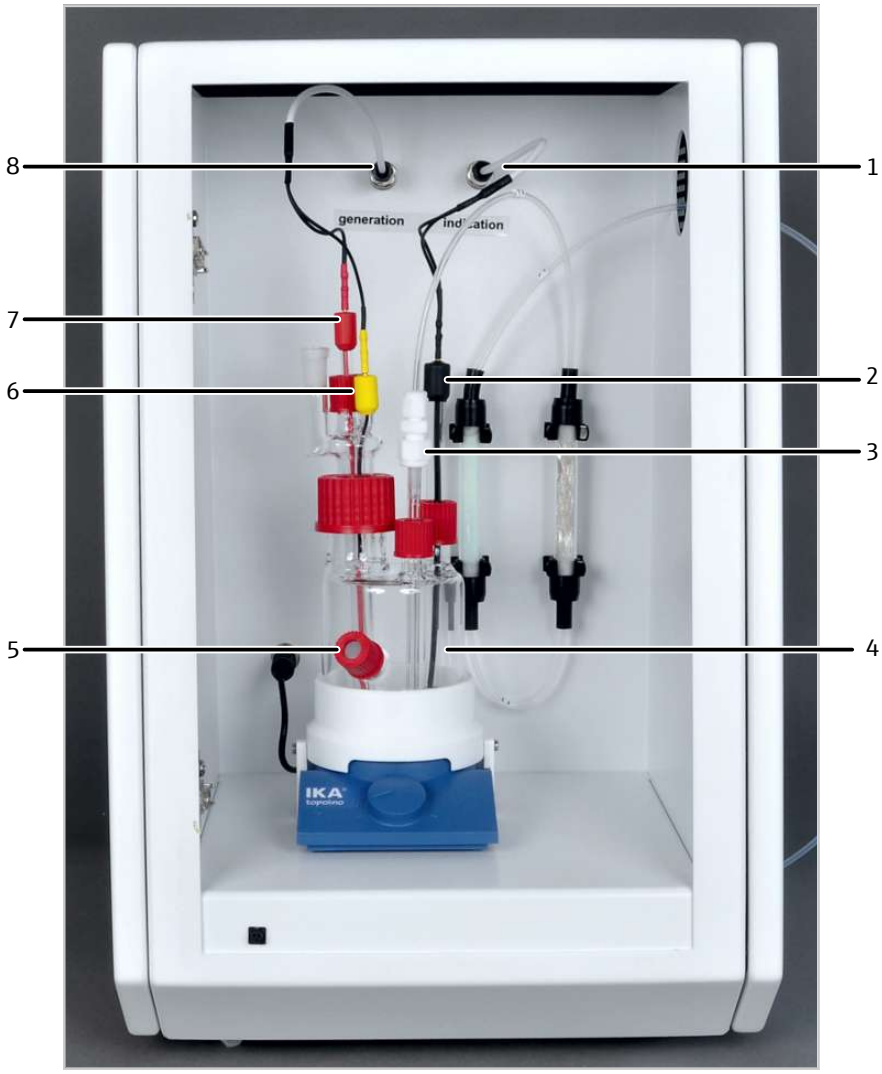


Fig. 53 Coulometric sulfur detector with measuring cell (without door)

1 Indicator electrodes connection
2 Indicator electrode (black)
3 Gas inlet
4 Measuring cell
5 Port for manual dosing
6 Anode (yellow)
7 Cathode (red)
8 Generator electrodes connection

10.3 Operation

10.3.1 Preparing the measuring cell

Preparing the measuring cell includes the following steps:
- Preparing the electrolyte solution
- Performing the end point routine

Two further solutions are required for the end point routine and for the check of the measuring cell. The creation of these solutions is also described in the following.

Preparation of the electrolyte solution

WARNING

Risk of chemical burns

100 % acetic acid (glacial acetic acid) can cause severe chemical burns. Gases may develop during shaking.

- Wear protective clothing when preparing the electrolyte solution. Work under an extractor.
- Observe all instructions and specifications in the safety data sheet.

⇨ Prepare the electrolyte solution one day before its use.

⇨ Reagents required: 10 ml acetic acid 100 % (glacial acetic acid), 10 g sodium acetate, 5 g potassium iodide, 7.5 g potassium chloride

▶ Dissolve the given amounts of sodium acetate, potassium iodide and potassium chloride in a 1-liter volumetric flask. Wait until the salts have completely dissolved.

▶ Carefully add 10 ml of glacial acetic acid. Keep moving the flask while pouring the acid. Carefully shake the solution.

▶ Fill the volumetric flask with ultrapure water up to the mark. Carefully shake the solution.

▶ Fill the measuring cell with approx. 100 ml of electrolyte solution (to the level of the port for manual metering).

✓ The electrolyte solution has been prepared and added to the measuring cell.

Performing the end point routine

A sodium sulfite solution (1000 mg/l) is required for the end point routine:

⇨ Reagents required: 393.9 mg sodium sulfite

▶ Fill a 100 ml volumetric flask with the specified quantity of sodium sulfite.

▶ Fill the volumetric flask with ultrapure water up to the calibration mark. Shake the flask until the sodium sulfite has completely dissolved.

Storage and handling of the sodium sulfite solution:
- The solution is sensitive to atmospheric oxygen and may thus be stored in a refrigerator for no longer than 1 month.
- For the end point routine, only 2 – 10 µl of the master solution are required.
- For better metering, diluted solutions can be used.

For the end point routine, the cell potential is set to the optimum operating range of the measuring cell.
- Operating range: 110 to 160 mV
- Optimal operation range: 120 mV

As soon as the cell potential goes outside of the operating range, the software will automatically start an end point routine.

Cell potential is lower than the operating range:
- Automatic generation start (iodine generation)

Cell potential is higher than the operating range:
- Add sodium sulfite solution when the software instructs.
- Titration runs automatically until the cell potential sinks to 120 mV.

An end point routine is also automatically started when electrolyte is replaced.

When working with a MMS, the addition of chloride ions to the measuring cells can be performed automatically if the indicator value exceeds the operating range while an analysis sequence is in progress. For this, the user must have prepared a suitable organic sulfur solution and placed it in the intended position (110) on the sample rack.

Checking the measuring cell

Only check the measuring cell if a defect of the detection module is suspected.

A sodium sulfate solution (1000 mg/l) is required for the check of the measuring cell.

⇨ Reagents required: 1.5482 mg sodium thiosulfate pentahydrate

▶ Weigh the indicated quantity of sodium thiosulfate into a 100 ml volumetric flask.

▶ Fill the volumetric flask with ultrapure water up to the calibration mark. Shake the solution until the salt has dissolved completed.

The solution can be kept for approx. 1 month in a tightly-sealed container.

Standard solutions can be prepared by diluting the solution.

100 µl of the diluted solutions contain the following TS amounts:
- 10 mg/l standard: 1 µg S absolute
- 100 mg/l standard: 10 µg S absolute

After the function test of the measuring cell with the sodium thiosulfate solution, the electrolyte must be replaced.

10.3.2 Operating the analysis system

▶ Place the measuring cell with electrodes and electrolyte solution in the detection module and connect it to electricity.

▶ Switch on the basic module and the detection module.

✓ The devices boot up. The status LED on the front of the basic module light up in green after approx. 30 s.

✓ The LED on the front of the detection module flashes during the run-in period. Depending on the detector, the run-in period can take up to 30 min. After this, the LED will light up continuously. Starting a measurement is only then possible.

▶ Open the gas supply and set the required gas pressure.

▶ Switch on the PC.

▶ Start the control and analysis software and login with your username and password.

▶ Initialize the analysis system by clicking on **[Initialize analyzer]**.

✓ The initialization and automatic detection of all connected components will be carried out.

▶ Ready the samples.

▶ Activate a pre-existing method via the **Method** | **Method - activate** menu item.

▶ Alternatively: Create a new method in the **Method** | **Method - new** menu. Select the measurement parameter in the method. Release and activate the method.

- Select **Start** | **Start - Analysis** in the menu.
- Select an analysis group or create a new one and confirm via **[OK]**.
- Create an analysis sequence.
- Enter the sample ID for all sample in the **Name** field.
- Release all sequence lines
- Confirm the entries with **[OK]**.
- Click the **[Start Measurement]** button.
  - ✓ The prepared analysis sequence is processed.

For manual sampling, follow the instructions in the software.

11 Carbon analysis with the C module 5100

11.1 Function and design

11.1.1 Function and measuring principle

Expansion of the basic module with the detection module allows determination of the carbon content in solid, liquid, paste-like, viscous and gaseous samples.

The detection module contains a wide-range NDIR detector. The carbon content in organic compounds can be determined as TC and EC/OC sum parameters with the detection module.

Carbon dioxide (CO_2) and water (H_2O) is created during thermal oxidation of the samples in the basic module. The gas mixture is dried and transferred to the NDIR detector.

$R + O_2 \rightarrow CO_2 + H_2O$

R: Hydrocarbon

The radiation sensor is CO_2-sensitive. The double bond between carbon (C) and oxygen(O) has a specific absorption range in the infrared wavelength band.

If a ray of light is projected through a cuvette arrangement, the CO_2 content in the measuring gas absorbs a proportion of the total radiation proportional to the CO_2 concentration.

11.1.2 Design

The detection module is used to determine the carbon content in solids, liquids and gases by measuring the IR IR absorption. All components required for determination are mounted inside the sealed housing.

The device door is closed tightly and cannot be opened. An LED is installed on the front of the detection module. The LED flashes during the run-in period and lights up permanently when the module is operational.

Fig. 54 Basic module with detection module and sampling module

The detection module can be used to determine TC and EC/OC.

- TC determination can be performed in both the multi-purpose combustion tube (standard tube) and the special EC/OC combustion tube.
- EC/OC determination requires the use of the EC/OC combustion tube.

Analytik Jena GmbH+Co. KG also provides special boats with hold-down clamps for EC/OC determination, with which, for example, diesel exhaust particles can be examined on quartz-fiber filters. The use of the ABD is required in horizontal operation mode.

11.1.3 Connection

The following components can be found on the rear of the device:

- Main switch, power connection and device fuses
- Inlet and outlet for the analysis gas
- Interface to the basic module

Fig. 55 Rear of the carbon detector

1 Equipment switch
2 Fuse holder
3 Power connection
4 Interface to the basic module
5 "Sample out" sample outlet
6 "Sample in" sample inlet

Communication with the basic module is performed via a 9 pin interface cable. The interface is labeled "C-NDIR".

The hose for the measuring gas coming from the basic module is connected to the "sample in" gas inlet. The gas outlet is labeled "sample out". The module does not need to be connected to the laboratory exhaust system.

11.2 Installation

NOTICE

Connecting or disconnecting electrical contacts may damage the sensitive electronic components of the basic module and of the detection module.

- Always connect the modules to power when they are switched off.

- Place the detection module to the left of the basic module. In the event of a series of detection modules: place the detection module to the left or right of the others.
- Connect the supplied power cable to the power cable connection on the rear of the module and to an earthed socket. Observe the permissible maximum voltage!
- Connect the detection module with the basic module via the interface:
  "C-NDIR" interface on the rear of the detection module
  "C-NDIR" on the rear of the basic module.
- Connect the measuring gas hose of the basic module to the "sample in" gas inlet on the rear of the module.
- Leave the "sample out" outlet unconnected or connect it to the measuring gas inlet of the next detection module.
  - ✓ Connection of the detection module is complete.

11.3 Operating the analysis system

- Switch on the basic module and the detection module.
  - ✓ The devices boot up. The status LED on the front of the basic module light up in green after approx. 30 s.
  - ✓ The LED on the front of the detection module flashes during the run-in period. Depending on the detector, the run-in period can take up to 30 min. After this, the LED will light up continuously. Starting a measurement is only then possible.
- Open the gas supply and set the required gas pressure.
- Switch on the PC.
- Start the control and analysis software and login with your username and password.
- Initialize the analysis system by clicking on **[Initialize analyzer]**.
  - ✓ The initialization and automatic detection of all connected components will be carried out.
- Ready the samples.
- Activate a pre-existing method via the **Method** | **Method - activate** menu item.
- Alternatively: Create a new method in the **Method** | **Method - new** menu. Select the measurement parameter in the method. Release and activate the method.
- Select **Start** | **Start - Analysis** in the menu.
- Select an analysis group or create a new one and confirm via **[OK]**.
- Create an analysis sequence.
- Enter the sample ID for all sample in the **Name** field.
- Release all sequence lines

- Confirm the entries with **[OK]**.
- Click the **[Start Measurement]** button.
  - ✓ The prepared analysis sequence is processed.

For manual sampling, follow the instructions in the software.

12 Carbon analysis with the TOC module 5100

12.1 Function and design

12.1.1 Function and measuring principle

The detection module contains a wide-range NDIR detector. Expansion of the basic module with the detection module allow determination of the following sum parameters:

Sum parameters	Samples	Basic module configuration
TC	Organic liquids, solids and gases	Vertical/horizontal operation
EC/OC	Elemental and organically bound carbon from particulate emissions (fine particulate matter, diesel engine exhaust, smoke gas	Horizontal operation with: ■ Special combustion tube for EC/OC determination
TC, TOC, NPOC, TIC	Water samples	Vertical operation with: ■ TOC combustion tube ■ Condensation coil

Carbon dioxide (CO_2) and water (H_2O) is created during thermal oxidation of the samples in the basic module. The gas mixture is dried and transferred to the NDIR detector.

$R + O_2 \rightarrow CO_2 + H_2O$

R: Hydrocarbon

The radiation sensor used in the TOC module is CO_2-sensitive. The double bond between carbon (C) and oxygen(O) has a specific absorption range in the infrared wavelength band.

If a ray of light is projected through a cuvette arrangement, the CO_2 content in the measuring gas absorbs a proportion of the total radiation proportional to the CO_2 concentration.

A proportion of the water sample is dosed manually into the TIC reactor to determine the inorganically bound carbon (TIC) content. The sample reacts with phosphoric acid in the TIC reactor. This creates CO_2, which is determined via the NDIR detector.

The detection module determines the CO_2 concentration multiple times per second and generates an integral over time from the signals. The integral is proportional to the carbon concentration in the sample.

12.1.2 Design

All components of the module to be operated or serviced by the user can be access via the door on the front of the module.

The module consists of the following main components:
- TIC condenser unit (with TIC reactor, gas-liquid separator, measuring gas dryer)
- Condensate pump
- Halogen trap and water traps for drying and cleaning the measuring gas.
- NDIR detector (in the rear part of the detector)
- Indicator and control elements, connections

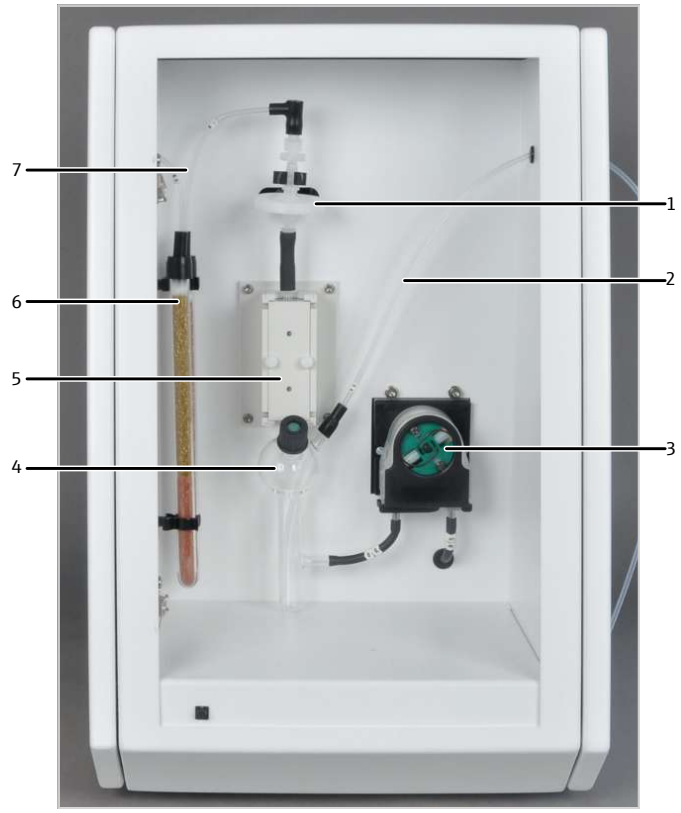


Fig. 56 TOC detector, door opened

1 Water traps
2 Measuring gas hose from the basic module (hose 80)
3 Condensate pump
4 TIC reactor
5 Cooling block (measuring gas dryer)
6 Halogen trap
7 Hose 81

The basic module must be equipped with the following components to analyze aqueous samples:

- TOC combustion tube
- Condensation coil

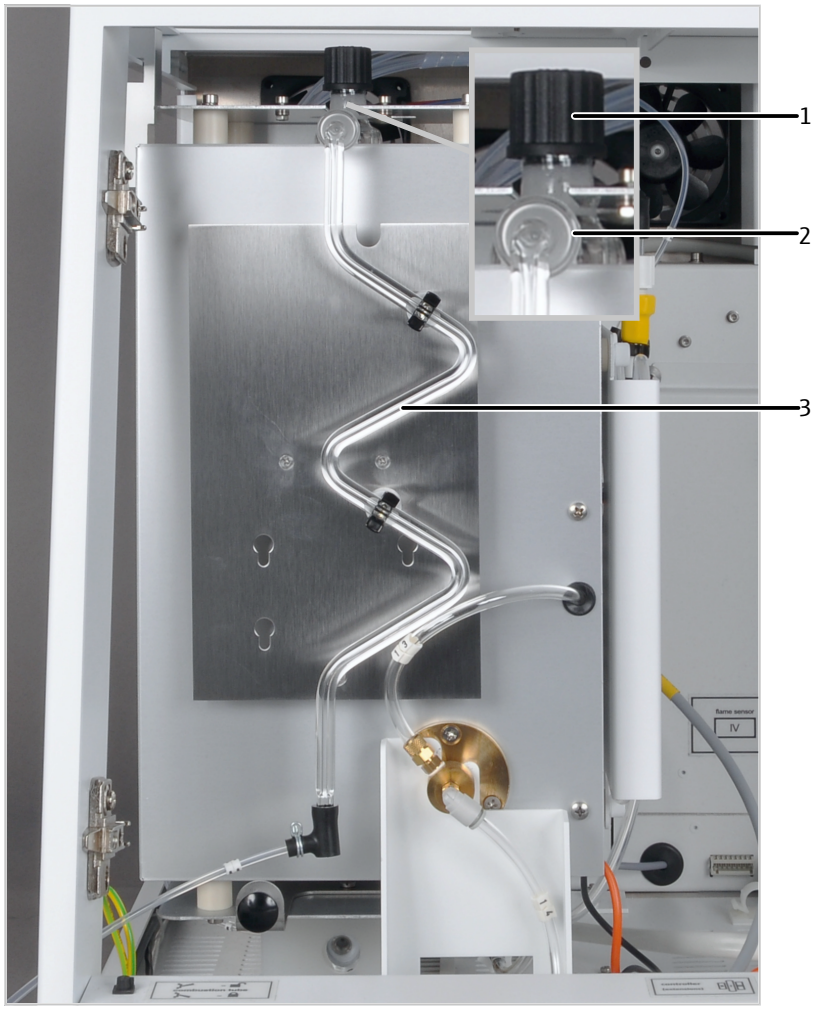


Fig. 57 Components in the basic module

1 TOC combustion tube injection port
2 Spherical joint (fasten with forked clamp)
3 Condensation coil

TOC combustion tube

The TOC combustion tube (reactor) is used to determine the TC, TOC and NPOC parameters in water samples. The combustion tube is made of quartz glass and is filled with a catalyst and additives. If the effectiveness of the catalyst decreases, the combustion tube has to be filled again.

Fig. 58 TOC combustion tube (unfilled)

Screw the screw cap with the septum on the top opening of the combustion tube. Connect the condensation coil with the aid of a fork type clamp to the spherical joint on the lateral outlet.

The oxygen gas supply (hose 3 of the basic module) is connected via a FAST connector at the lateral outlet directly under the screw cap. The tube holder is used to fasten the TOC combustion tube in the furnace.

Fig. 59 Tube holder for the TOC combustion tube

Sampling

Water samples are dosed directly into the TOC combustion tube via the injection port with microliter syringes. A syringe with scale is used for manual dosing. For automatic sampling via the autosampler, special microliter syringes are used. The syringes have special dimensions and therefore have no scale. They are unsuitable for manual operation for this reason. The syringes have a gas connection for analyses in NPOC mode. The injection volume is: 50 to 500 µl. Optimum measurement results are achieved when 50 to 100 % of the volume of the microliter syringe is used.

The injection port is equipped with temperature-resistant septums with a high penetration tolerance.

A proportion of the sample is dosed directly into the TIC reactor of the TOC module with a microliter syringe with a scale to determine the inorganic carbon content (TIC). Only manual dosing is permitted for this.

Measuring gas drying and cleaning

For the analysis of water samples, the basic module is equipped with a condensation coil made of glass. The condensation coil is connected to the TOC combustion tube via the spherical joint. Connect hose 80 to the other end of the condensation coil via the the FAST connector.

The measuring gas cools quickly in the condensation coil and the water vapor condenses. The mixture of measuring gas and water is transferred to the TIC reactor in the detection module via hose 80.

Fig. 60 Condensation coil

1 FAST connector 2 Spherical joint

The detection module is equipped with the TIC condensation unit. The TIC condensation unit consists of the following components:

Component	Task
TIC reactor	TIC determination
Gas-liquid separator	Separation of the liquid phase (condensate, waste solution from TIC determination)
Cooling block	Water vapor condensation (measuring gas drying)

The mixture of measuring gas and water is added via the lateral outlet of the TIC reactor via hose 80.

40 % phosphoric acid is placed in the TIC reactor to determine TIC content. The acid and the sample are added manually via the front connection with septum.

The measuring gas is routed via the top connector in the direction of the water traps from the TIC condensation vessel.

The condensate or waste solution from TIC determination is pumped out via the lateral outlet on the glass container. The condensate pump transports the waste to the "waste" outlet on the rear of the device.

The two following components protect the detector and remove disruptive substances from the measuring gas.

- Water traps
- Halogen trap

Two water traps are installed in the TOC module. They remove water from the measuring gas. The water traps are connected to the measuring gas outlet of the TIC reactor.

The water traps prevent condensed water from being transported into the path of the measuring gas after leaving the TIC condensation unit. The larger water trap (TC prefilter) retains aerosols during operation. The smaller water trap retains rising water (disposable retention filter).

The halogen trap removes gaseous halogen compounds from the measuring gas. The halogen trap is installed in the measuring gas path downstream of the water traps. The U-tube is filled with special copper wool. The filling of the halogen trap must be replaced at the latest when half of the copper wool has turned black.

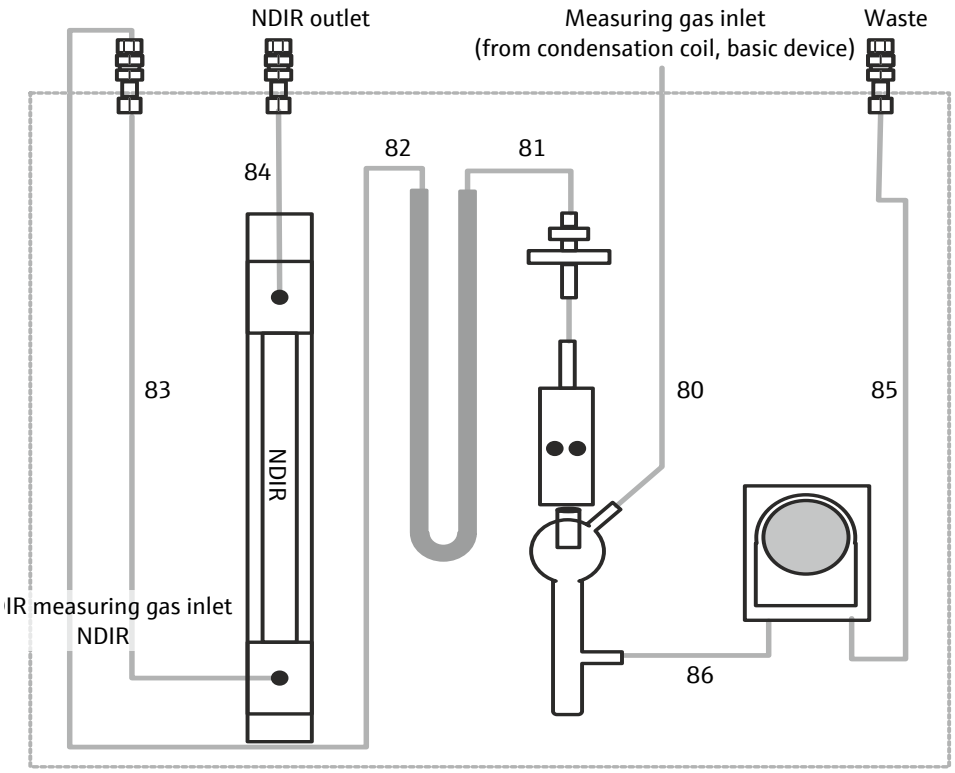


Fig. 61 Hose diagram of the TOC detector

Straight and angled FAST connectors are used to fasten hose connections.

12.1.3 Connection

The device switch for switching the detection module on and off is located on the top right of the rear of the device (when viewed from the front). The device fuse and power connection are located beneath it.

Fig. 62 Rear of the TOC detector

1 "External (out)" connection socket (25-pin)
2 "C-NDIR" interface cable connection (9-pin)
3 "External (in)" interface cable connection (25-pin)
4 "Sample in" measuring gas inlet
5 "Sample out" measuring gas outlet
6 "Waste" waste outlet
7 Power switch, fuse holder, power connection
8 Holder for the NPOC purging hose

Communication with the basic module is performed via a 2 interfaces:
- The interface for the 9-pin interface cable is labeled "C-NDIR".
- The interface for the 25-pin interface cable is labeled "External (in)".

The "External (out)" interface is not used.

The hose connection are required to be factory-prepared for TOC measurement (in water samples) The "Sample in" measuring gas inlet is connected with hose 82.

The purged condensate or waste solution from TIC determination is drained via the "waste" outlet on the rear of the module. For this, a waste hose is connected to the connector "waste" and inserted into a waste container (included in the scope of delivery).

NPOC

The connection for the NPOC purging gas is located on the rear of the basic module and is labeled with "out ABD". The purge hose is connected to the connector via a FAST connector. The purge hose is fastened to the holder on the rear of the detection module with the provided connector. From there, a hose with an outer diameter (AD) of 1.6 mm continues to the holder on the sampler. For manual sample preparation, the hose can be immersed directly in the sample.

TC, EC/OC

The detection module can be used to determine TC and EC/OC.

TC determination can be performed in both the multi-purpose combustion tube (standard tube) and the special EC/OC combustion tube.

EC/OC determination requires the use of the EC/OC combustion tube.

For the connection to the basic module, hose 82 is removed from the "sample in" measuring gas inlet on the rear of the detection module. The "sample in" measuring gas inlet is connected to the "Sample OUT N/S/C" outlet on the basic module.

12.2 Installation

NOTICE

Connecting or disconnecting electrical contacts may damage the sensitive electronic components of the basic module and of the detection module.

- Always connect the modules to power when they are switched off.

- Place the detection module to the immediate left of the basic module.
- Connect the supplied power cable to the power cable connection on the rear of the module and to an earthed socket. Observe the permissible maximum voltage!
- Connect the detection module to the basic module via the two interfaces:
  - 9-pin interface cable
    "C-NDIR" interface on the rear of the detection module
    "C-NDIR" interface on the rear of the basic module
  - 25-pin interface cable
    "external (in) interface on the rear of the detection module
    "External" interface on the rear of the basic module
- Install the halogen trap and the water traps in the detection module as shown in the illustration. Connect both with hose 81.
- Install the TIC reactor. Connect the TIC reactor to the water traps. Connect the TIC reactor to the condensate pump via hose 86.
- Connect the measuring gas hose (hose 80) from the basic module to the lateral outlet of the TIC reactor.
  Hose 80 is connected to the condensation coil outlet in the basic module later.
- Place the waste canister to the left of the detection module.
- Connect the waste hose to the "waste" outlet on the rear of the detection module. Route the waste hose into the waste canister.
- Leave the "sample out" outlet unconnected or connect it to the measuring gas inlet of the next detection module.

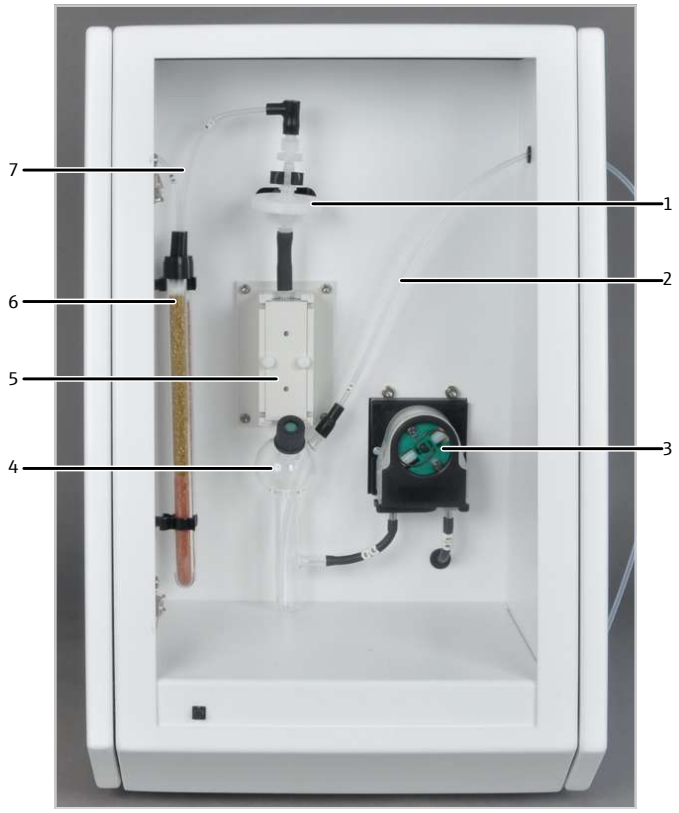


Fig. 63 TOC detector, door opened

1 Water traps
2 Measuring gas hose from the basic module (hose 80)
3 Condensate pump
4 TIC reactor
5 Cooling block (measuring gas dryer)
6 Halogen trap
7 Hose 81

Preparing the basic module

- Fill the TOC combustion tube as shown in the illustration.
- Insert the TOC combustion tube into the combustion furnace of the basic module. Connect the oxygen supply (hose 3). Fasten the combustion tube with the tube holder.
- Install the condensation coil in the basic module. Connect the condensation coil with the TOC combustion tube with the spherical joint. Secure the spherical joint connection with a clamp.
- Connect the condensation coil to the TIC reactor in the detection module with hose 80. Route the hose through the side openings of the modules for this.

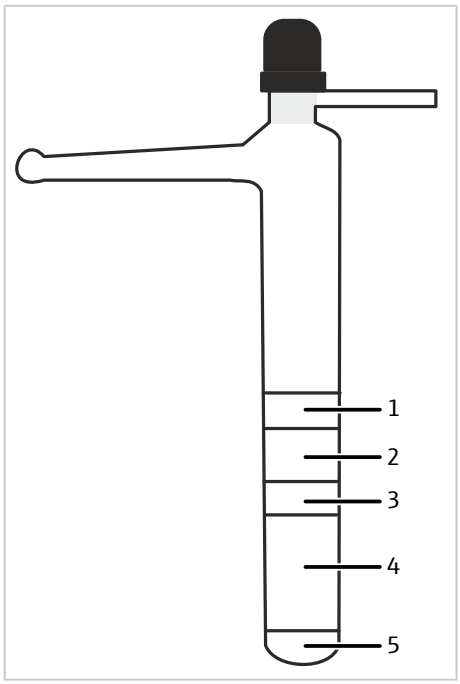


Fig. 64 TOC combustion tube

1 HT mat
2 Quartz glass fragments
3 Quartz wool
4 Catalyst
5 Quartz wool

For NPOC determination

- Prepare the detection module and the basic module as previously described.
- Connect the NPOC purge hose (hose 16) to the "out ABD" connection on the basic module.
- Connect the purge hose with a hose with an outer diameter (AD) of 1.6 mm. Fasten the hose to the rear on the TOC module.
- Route the hose to the holder on the sampler. Alternatively, use the hose for manual purging of the acidified samples.

For TC, EC/OC determination

The TOC module can be configured for TC determination with the basic module and the multi-purpose combustion tube or the special combustion tube for EC/OC determination. For EC/OC determination, the special combustion tube for EC/OC determination must be used. The measuring gas is routed directly from the combustion tube into the NDIR detector via the "sample in" outlet.

- Remove hose 82 from the "sample in" measuring gas inlet on the rear of the detection module.
- Connect the "sample OUT N/S/C" outlet on the basic module with the "sample in" measuring gas inlet with hose 9.

12.3 Operation

12.3.1 Operating the analysis system

- Switch on the basic module and the detection module.
  - ✓ The devices boot up. The status LED on the front of the basic module light up in green after approx. 30 s.

✓ The LED on the front of the detection module flashes during the run-in period. Depending on the detector, the run-in period can take up to 30 min. After this, the LED will light up continuously. Starting a measurement is only then possible.

- Open the gas supply and set the required gas pressure.
- Switch on the PC.
- Start the control and analysis software and login with your username and password.
- Initialize the analysis system by clicking on **[Initialize analyzer]**.
  ✓ The initialization and automatic detection of all connected components will be carried out.
- Ready the samples.
- Activate a pre-existing method via the **Method** | **Method - activate** menu item.
- Alternatively: Create a new method in the **Method** | **Method - new** menu. Select the measurement parameter in the method. Release and activate the method.
- Select **Start** | **Start - Analysis** in the menu.
- Select an analysis group or create a new one and confirm via **[OK]**.
- Create an analysis sequence.
- Enter the sample ID for all sample in the **Name** field.
- Release all sequence lines
- Confirm the entries with **[OK]**.
- Click the **[Start Measurement]** button.
  ✓ The prepared analysis sequence is processed.

For manual sampling, follow the instructions in the software.

12.3.2 Notes for measuring operations

Observe the following instructions for operation of the TOC detector:

- Regenerate the TIC reactor before performing TIC determination (→ "Regenerating the TIC reactor" 🖹 159).
- Dilute highly acidic and saline samples, e.g., 1:10.
  Aerosols can form in the TIC reactor during analysis of these samples. The capacity of the halogen trap will then be exhausted quickly and may clog.
- The water traps protect the NDIR detector from aerosols. In the event of excessive aerosol formation, the software interrupts the carrier gas supply. Also in the event of excessive aerosol formation, interrupt the connection between the water traps and the outlet of the TIC reactor.
- To acidify samples: Use hydrochloric acid (HCl) at c = 2 mol/l. Create the acid from concentrated HCl (p.a.) and TOC water.
- For TIC determination: Use 40 % orthophosphoric acid (H_3PO_4). Create the acid from concentrated orthophosporic acid (p.a.) and TOC water.
- Only store solutions in clean, particle-free glass containers (volumetric flasks, sample containers).

Carbon dioxide and organic vapor in the air of the laboratory can slightly alter the concentration of samples and standard solutions.

- Remove the source of any organic vapor from the laboratory.
- Create low-concentration solutions (c < 1 mg/l) under a fume hood.
- Keep the free space above the liquid small in containers.
- Cover the sample containers with foil for sampler operation (differential mode).

13 Troubleshooting

13.1 General notes

For fault analysis, log files can be recorded. Log file recording should be activated after consultation with Analytik Jena GmbH+Co. KG customer service for specific faults.

The save location of the log files can be defined via the **Extras** | **Configuration** menu item in the **Configuration** | **Error analysis** window.

CAUTION

- If faults cannot be remedied by the customer, the Analytik Jena GmbH+Co. KG service department must always be informed. This also applies for the repeated occurrence of individual faults.
- Send the correspond files to the service department via email for fault diagnosis (address on inside of the front cover).

13.2 Remedying software message faults

Communication problems between the hardware and the software can often be remedied by a basic initialization of the measuring system (→ "Initializing the basic module and the system components" 104).

NOTICE

Communication fault due to wrong USB cable

- Use the cable supplied by Analytik Jena GmbH+Co. KG.
- Extensions are not permitted for the USB connection!

Error code	Error message/cause	Remedy
1	No response from firmware!	
	Basic module not switched on	Switch on the basic module
	Basic module disconnected from PC	Check the connection between the basic module and the PC
	Incorrect port selected	Check which port the device is plugged into on the PC Select another port (**Configuration** | **Interface** menu item) Initialize

Error code	Error message/cause	Remedy
2	Serial port not available!	
3	Serial port not reachable!	
	communication problems	Disconnect the USB connection between the basic module and the PC and reconnect after approx. 10 s. Initialize
7	Operating system faults: Unauthorized access	
	Undefined multiWin end	Exit the software and switch off the device Detach the USB cable and re-connect after approx. 10 s Restart the operating system (PC) Switch on the device Restart the software
12	Signal echo received, check port selection	
	Incorrect port selected	Check port selection
14	Data transfer interrupted	
	No data transfer for 10 s	Check port selection
17	Incorrect interface protocol ID	
	Fault after update (the program versions of the firmware and multiWin do not match)	Update required
20	Timeout: InitEnd	
	Timeout during initialization	Initialize
21	Timeout: StatusBusy	
	Timeout during operation (device not ready to measure)	Acknowledge message Initialize
22	Timeout: End	
	Timeout when exiting multiWin	Acknowledge message Initialize
23	Timeout: StopEnd	
	Timeout during measurement cancellation	Acknowledge message Initialize
50	Firmware reset	
	Internal computer (firmware) restarted	Acknowledge message Initialize
61	Command from PC incomplete	
62	Command from PC without STX	
64	Command from PC CRC error	
65	Command from PC invalid	
66	Command from PC invalid MEAS command	
	Communication error	Acknowledge message Initialize
100	C sensor: No connection	
101	C sensor: CRC error	
	Communication interrupted after sensor detection during initialization	Acknowledge message Initialize

Error code	Error message/cause	Remedy
104	C sensor: Analog values outside range	
	The analog values of the detector are outside the operating range	Check the quality of the carrier gas Initialize Check the analog values in the component test (**System** | **Component test** | **NDIR** menu item)
110	N sensor: No connection	
120	S sensor: No connection	
130	Cl sensor: No connection	
	Communication interrupted after sensor detection during initialization	Acknowledge message Initialize
131	Cl sensor: Incorrect command structure	
	Communication to the chlorine module interrupted	Acknowledge message Switch the chlorine module on/off Initialize
132	Cl sensor Indicator error	
	Indicator value out of range after titration start (measurement cannot start)	Acknowledge message Initialize Run an end point routine Check the status of the titration cell (**System** | **Component test** | **Chlorine** menu item)
133	Cl sensor: Incorrect cell	
	No initialization after cell change	Initialize
134	Cl sensor: Incorrect status	
	Communication error	Acknowledge message Initialize Check the status of the titration cell (System | Component test | Chlorine menu item)
135	Cl sensor: Incorrect version	
	Transmission fault	Acknowledge message Initialize Check the status of the titration cell (System | Component test | Chlorine menu item)
200	Gas box: No connection	
	Communication error	Acknowledge message Initialize

Error code	Error message/cause	Remedy
201	Gas box: Fault when setting the target flow	
	Communication to gas box interrupted	Acknowledge message Initialize
202	Gas box: Conversion fault 1	
203	Gas box: Conversion fault 2	
204	Gas box: Conversion fault 3	
205	Gas box: Conversion fault 4	
	Communication faulty (readout of flows from gas box faulty)	Acknowledge message Initialize
206	Gas pressure error	
	Overpressure in the analysis system due to blocked gas paths	⚠ WARNING! Extreme caution is required during system overpressure! Never switch off a device subject to overpressure! Otherwise, risk of injury to operating personnel and damage to the analysis system is present. Follow the instructions in the section on (→ "Behavior during overpressure faults (0206, gas pressure fault)" 14)
220	Sampler: no connection	
	Communication interrupted after autosampler detection during initialization	Acknowledge message Initialize
222	Boat: Broken	
	Boat defect during removal from the combustion tube (only if boat sensor is used)	Remove the broken boat from the system Initialize
223	Sampler: Incorrect peak value	
	No syringe inserted	Insert syringe in autosampler Initialize
	Dosed volume in the method greater than inserted syringe volume	Adjust dosed volume or insert suitable syringe Initialize
	A method for dosing liquids is to be activated and the gripper and solids track are still inserted	Insert sample rack for liquid samples Insert syringe Initialize
224	Sampler: Incorrect gripper	
	No gripper inserted	Insert gripper in autosampler Initialize
	The method for solids is to be enabled and the syringe and sample rack for liquid samples are still inserted	Insert solids rack Insert gripper Initialize
226	Sampler: Runtime exceeded	
	Completion message for the autosampler movement takes too long (autosampler faulty)	Record log files Inform customer service department

Error code	Error message/cause	Remedy
230	ABD: No connection	
	Communication error after ABD was detected during initialization	Acknowledge message Initialize
231	ABD: Runtime exceeded	
	Completion message for the ABD movement takes too long	Check that the flame sensor is properly applied and connected Record log files Inform customer service department
232	Flame sensor error	
	Flame sensor calibration failed	Record log files Inform customer service department
250	LPG: No connection	
	Communication interrupted after LPG detection during initialization	Acknowledge message Initialize
251	LPG: Runtime exceeded	
	Communication error Completion message at dosing end not received	Acknowledge message Check argon gas inlet pressure Initialize
252	LPG: Argon missing during dosing	
	No argon applied to LPG module	Check gas supply Check gas inlet pressure
253	LPG: Incorrect sample volume	
	Dosed volume is not a whole multiple of the sample loop inserted	Adapt dosing volume to sample loop volume
260	Sample handling missing	
	No sampler module detected	Connect at least one sampler module Initialize
270	Auto-injector syringe: No connection (applies to Autoinjector, Autoinjector AI-EA)	
	No communication with auto-injector	Acknowledge message Initialize
271	Auto-injector syringe: Runtime exceeded (applies to Autoinjector, Autoinjector AI-EA)	
	Communication error Completion message at dosing end not received	Acknowledge message Check auto-injector Initialize
272	Auto-injector syringe: Incorrect syringe size	
	Autoinjector: Dosing volume and syringe size different	Adapt the dosing volume and/or the syringe size
	Autoinjector AI-EA cannot complete this order	Initialize

Error code	Error message/cause	Remedy
273	Auto-injector syringe: Syringe drawn incorrectly (Autoinjector only)	
	Syringe not drawn back all the way	Draw syringe back all the way Insert syringe
274	Autoinjector: No connection	
	Auto-injector coupling not found	Check connection Acknowledge message Initialize
	No Autoinjector AI-EA was found	
	Autoinjector AI-EA not connected or faulty	Check connection Acknowledge message Initialize
275	Autoinjector: No syringe detected	
	Syringe not drawn Syringe detection faulty	Repeat sampling with drawn syringe Test a different syringe
300	Temperature controller: No connection	
	Communication error	Acknowledge message Initialize
304	Temperature controller: Communication error	
	Temperature cannot be set	Acknowledge message Initialize
400	Syringe pump: No connection	
	Communication error	Acknowledge message Initialize
401	Syringe pump: Initialization	
402	Syringe pump: Invalid command	
403	Syringe pump: Invalid operand	
404	Syringe pump: Invalid command sequence	
407	Syringe pump: Device not initialized	
	Communication error Syringe pump faulty	Acknowledge message Search for cause and remedy fault Initialize
409	Syringe pump: Pump sluggish	
	Gas hose clogged or kinked Syringe pump faulty	Acknowledge message search for cause and remedy fault Initialize
410	Syringe pump: Valve sluggish	
	Syringe pump faulty Valve faulty	Acknowledge message Search for cause and remedy fault Initialize

Error code	Error message/cause	Remedy
411	Syringe pump: Pump step not permitted	
415	Syringe pump: Command error	
420	Syringe pump: incorrect type	
	Communication error	Acknowledge message Initialize

13.3 Initializing the basic module and the system components

Initialization of a measuring system establishes communication between the measuring system and the computer. The multiWin program differentiates between a standard initialization and a basic initialization.

With standard initialization, only the system components active before the last shutdown of multiWin are queried and the method last active is loaded.

The basic initialization, on the other hand, is more thorough and tests all connected system components activated in the multiWin program in the window. The basic initialization must always be performed in the following situations:

- Connection of new system components
- Recognition of system components which were shut down or were not connected during the last initialization
- Fault in the communication between the measuring system and the computer

Performing the basic initialization

Basic initialization is always performed if the **Device - edit** window was opened and exited via **[OK]**:

- Select the **Device** | **Device - edit** menu item.
- Make any necessary changes and exit the **Device - edit** window via **[OK]**.
- Click on **[Initialize analyzer]** in the main window.
  - ✓ The system is initialized and the method last used is activated. If the initialization was successful, the **[Start Measurement]**, **[Activate method]** and possibly the **[Start calibration]** buttons are displayed in the main window.

Standard initialization

Click on the **[Initialize analyzer]** button in the main window. Alternatively, select the **System** | **Initialize** menu item.

13.4 Displays in the window Status analyzer

13.4.1 Overview

In the **Status analyzer** window, information on the device status or information on individual modules is displayed.

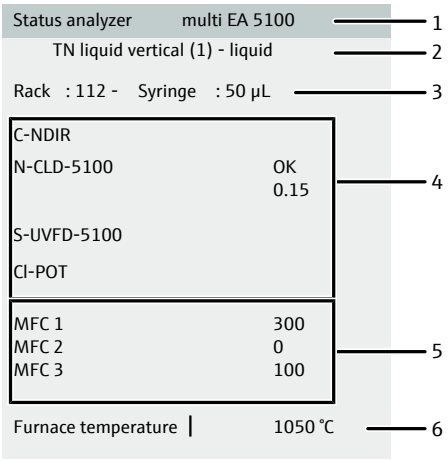


Fig. 65 Status analyzer window

1 Window name and basic module
2 Active method and sample state
3 Sampling modules
4 Detectors with sensor values
5 Gas flow displays
6 Furnace temperature (horizontal line – horizontal installation, vertical line – vertical installation)

The displays in the **Status analyzer** window are color-coded. The colors have the following meanings:

Color	Description
Black	Status of the corresponding component OK, device ready for measurement
Gray	Detector is inactive
Green	Detector OK, device ready for measurement (OK) Or Detector busy, measurement can only be started once the routine is complete (detector-specific)
Red	Component not ready for measurement: ■ Run-in period not yet complete; wait for run-in time to complete ■ Error: Troubleshooting, access the corresponding information for the component via the **System** | **Component test** menu item.

13.4.2 Method

The following can be displayed in the top line of the **Status analyzer** window:

Display	Description
TN(1) - liquid	Example of a method: Name(version) – state Possible states: ■ Liquid ■ Solid ■ GSS ■ LPG ■ AOX, AOX solid ■ EOX liquid, EOX solid
No method displayed (display empty)	Device not ready for measurement, no method active: Activate method

13.4.3 Sampling modules

Example	Rack: 112 - syringe: 50 µl
Meaning	Sampling module - rack - syringe size

All sampling modules detected during initialization are displayed and (possibly) described in detail. The following displays are possible:

Display	Description
GSS (pressureless)	Gas dosing module for sampling from bag or in combination with the GSS adapter box from sample cylinder
LPG	LPG module
GSS/LPG	LPG/GSS combination module for sampling under pressure
Rack: 112 - syringe: 50 µl	Autosampler: Specification of rack size and syringe size
Rack: 35 - gripper	Autosampler: Specification of rack and gripper
Auto-injector - syringe: 50 µl	Auto-injector type and syringe size
ABD	Automatic boat feed
ABD - FS	Automatic boat feed and flame sensor

The following status displays are possible:

Device not ready for measurement	
Display	**Description**
No sampling module displayed (display empty)	Device not ready for measurement, no sampling module identified: ■ Connect and activate at least one sampling module ■ Basic initialization
Auto-injector - syringe: ?	No auto-injector syringe detected ■ Initialize ■ Insert auto-injector syringe and register it in multiWin ("Extras | New auto-injector syringe" menu item)

13.4.4 Detectors

Example	CI-POT OK	3050
Meaning	Detector status	Current sensor value

All detector modules detected during initialization are displayed here. The following displays are possible:

Display	Description
C-NDIR	C module 5100 or TOC module 5100 with NDIR detector
N-CLD	N module 5100 with chemiluminescence detector
S-UVFD	S module 5100 basic and S module 5100 MPO with UV floures-cence detector
S-Coul	S module 5100 coulometric with microcoulometer
CI-POT	CI module 5100 with "high sensitive" measuring cell
CI-AMP smallCell	CI module 5100 with "sensitive" measuring cell
CI-AMP largeCell	CI module 5100 with "high concentration" measuring cell

The respective status of the detector module is indicated by the color:

Display	Description
Black	Detector is active, status is queried and displayed (see example above)
Gray	Detector is inactive, status is not displayed
Green	Detector OK, device ready for measurement (OK) Or Detector busy, measurement can only be started once the routine is complete (detector-specific)
Red	Error, see overview below

The following status displays are possible:

Device ready for measurement	
Display	**Description**
OK (green, black)	Detector is ready for measurement
Device not ready for measurement - general	
Display	Description
No detector displayed (display empty)	No detector detected ■ Activate detector ■ Basic initialization
Communication error (red)	Communication interrupted: ■ Deactivate/activate the device ■ Basic initialization
No connection (red)	Connection interrupted: ■ Check connection cable ■ Deactivate/activate the device ■ Basic initialization
Device not ready for measurement – Cl-POT	
Display (red)	**Description**
Inactive	No cell detected: ■ Insert cell ■ Basic initialization
Drift exceeds range	The current indicator drift is greater than the maximum positive drift set in the method or the max. negative drift defined in the system. ■ Wait for the drift to go back into range (this is normal immediately after cell maintenance or after commissioning the device) ■ Check the maximum drift setting in the method and increase as necessary (e.g., to 100 Counts/min). ■ Access further values via the **System** | **Component test** | **Chlorine** menu item. ■ If the drift remains outside of range but is completely stable, the following is also possible: Setting the value "1" for drift shuts off drift monitoring.
End point routine required	Indicator value outside the operating range of the titration cell: ■ Indicator value greater than 10000: End point routine starts automatically ■ Indicator value less than 1000: Start an end point routine via the **System** | **End point routine** menu item and follow the instructions
Cell temperature exceeds range	Current cell temperature does not correspond to cell temperature configured in the method: ■ Wait until the desired cell temperature is achieved ■ Access the values via the **System** | **Component test** | **Chlorine** menu item. ■ Check the cell temperature settings for the method and adjust as necessary
Device not ready for measurement – Cl-POT	
Display (green)	**Description**
End point routine	Automatic end point routine running: ■ Wait for the end point routine to finish

Drift determination	Drift determination immediately after titration or end point routine: ■ Wait until drift determination is complete (approx. 1 minute)
Device not ready for measurement - CI-AMP	
Display (red)	**Description**
Inactive	No cell detected: ■ Insert cell ■ Basic initialization
Device not ready for measurement - CI-AMP	
Display (green)	**Description**
Stand-by titration	Interval titration running: ■ Measurement start possible
End point routine	End point routine running: ■ Wait for the end point routine to finish
Device not ready for measurement – C-NDIR	
Display (red)	**Description**
Analog values warning	Analog values outside range: ■ Access the values via the **System** | **Component test** | **NDIR** menu item.
Running-in time	Detector not yet ready for operation: ■ Wait for the run-in period after activation to complete (approx. 30 min)
Device not ready for measurement – S-UVFD or N-CLD	
Display (red)	**Description**
Preheating phase	Detector not yet ready for operation: ■ Wait for the run-in period after activation to complete (approx. 30 min)
Vacuum/pressure error	Pressure in the detector outside the permitted range: ■ See device faults of the N detector

13.5 Device faults on the basic module

Error	Possible cause	Remedy
Furnace does not heat	Thermocouple connector not connected	Connect the connector (→ "Removing and installing the combustion furnace" 🖹 139)
	Temperature set incorrectly in the software	Check temperature configuration in the method
	No method loaded	Load method
	Malfunction in power supply	Switch on the device Check the internal fuses Check the connection between the basic module and the PC
	Malfunction in the internal electronics	Inform customer service department
Furnace temperature is outside tolerance limits or target temperature is not reached	Temperature control faulty Electronics error	Inform customer service department

Error	Possible cause	Remedy
Process gases (inlet flow) not supplied	Gas supply not connected	Connect the gas supply
	Primary gas pressure too low	Set the gas inlet pressure to 600 kPa (6 bar) at the delivery point
	Gas supply leaking	Check gas supply
	No method loaded	Load method
	Gas box faulty	Inform customer service department
Target flow at the outlet to the detector too low	Connection between hose, angled adapter and combustion tube not correct	Check connection and ensure correct fit at the connection points
	Pneumatic seal in the coupling not sealing combustion tube	Check Ar supply Flip the toggle switch for the pneumatic seal down
	Septum incorrectly positioned in the injection port or leaking	Check position of the septum, insert new septum if necessary
	Connection of the membrane dryer or transfer line to the auto-protection valve assembly leaking	Check connections (do not jam thread, tighten finger-tight)
	Transition between coupling tube and ABD in horizontal mode leaking	Check the seal of the coupling tube Check the alignment of the combustion tube to the coupling tube Tighten screw finger-tight
Gas escaping from pneumatic seal (audible hiss)	Connector for hose 11 loose	Press hose 11 firmly into the quick-release connector
	Pneumatic seal defective	Replace pneumatic seal (→ "Maintenance of the auto-protection valve assembly" 🖹 129)
Auto-protection valve assembly not heating	Connector not connected	Connect the connector
	Heating faulty Temperature control faulty	Inform customer service department
Auto-injector not detected	Auto-injector and autosampler switched on at the same time	Switch off the autosampler

13.6 Analytical problems in the basic module

Error	Possible cause	Remedy
Burns at the cannula	Argon and oxygen connections on the combustion tube mixed up	Connect the process gases to the combustion tube properly
	Unsuitable settings in the method: Argon gas flow too low in the inlet (in particular for methods with gas autosamplers)	Adjust the method parameters to the analytical requirements
Low results independent of detection	Dosing fault	Check dosing by auto-injector or autosampler
	System leaking	Check system tightness
	Temperature set too low	Check temperature configuration in the method
	Wrong or unsuitable calibration	Check calibration, recalibrate as necessary
	Sample loss due to vaporization or spillage	Keep liquid samples closed. If possible, used a cooled sampler. Check sampler function for solids
	Post-combustion period insufficient	Particularly for solids, a post-combustion period of at least 120 s must be set.
	Sooting in the system	Clean or replace sooty components
Carryover	Inadequate sampler component rinsing	Rinse dosing syringes adequately prior to sampling
	Combustion tube not rinsed adequately	Rinse combustion tube adequately with clean solvent, i.e. blank measurements until values are constant
	Contamination of the injector head or the sample sluice	Replace septum Clean sluice
Scattering measurements	Dosing faulty	Check dosing
	Combustion tube contaminated or severely crystallized	Clean or replace combustion tube

13.7 Device faults on the N module 5100

Error	Possible cause	Remedy
LED in front panel flashing Ozone generator off	■ Run-in period of detection module not yet complete	■ Wait for the completion time for the run-in period of approx 30 min to elapse.
	■ Device in standby	■ Initialize the device
	■ Gas deactivated	■ Activate gas flow, see software manual
	■ Module not connected to basic module	■ Connect module (→ "Installation" 94)
	■ No method or method with no CLD active	■ Activate a method with nitrogen detection
	■ Other fault causes	■ Check the condition of the module under **System** | **Component test**
Pressure error	■ Gas outlet flow impeded	■ Check the "sample out" connection for unimpeded flow
	■ Absorber clogged	■ Replace absorber (→ "Replacing the absorber" 143)
	■ Converter worn out/old	■ Inform customer service department
	■ Pump faulty	■ Inform customer service department
	■ Device leaking or defective	■ Inform customer service department
Exhaust gas temperature outside range	■ Run-in period of detection module not yet complete	■ Wait for the completion time for the run-in period of approx 30 min to elapse.
	■ Thermal ozone decomposer defective	■ Inform customer service department
Heating temperature sensor defective	■ Thermal ozone decomposer defective	■ Inform customer service department
Smell of ozone	■ Chemical ozone decomposer old/ineffective	■ Replace chemical ozone decomposer (→ "Replacing the chemical ozone decomposer" 144)
	■ Gas hoses on the ozone generator leaky or loose	■ Check hoses ■ Search for leak as necessary with indicator paper
	■ Device leaking or defective	■ Inform customer service department

13.8 Analytical problems during TC determination

Error	Possible cause	Remedy
Scattering measurements	■ Oxygen supply to the module interrupted	■ Check oxygen connection, reconnect as necessary
	■ Absorber clogged or worn out	■ Replace absorber, see (→ "Replacing the absorber" 143)
	■ Sample unsuitable for vertical operation mode (droplet formation)	■ Use the horizontal operation mode
	■ In vertical operation mode: Quartz wool not present or at the wrong position in the combustion tube	■ Check the position of the quartz wool, adjust as necessary
	■ Sample evaporates before dosing	■ Use a cooled sampler.
	■ Sample too viscous too be drawn in without air pockets	■ Use the horizontal operation mode and dilute the sample, or dose directly as a solid.
	■ Inhomogeneous sample or sample containing particles	■ Homogenize sample
Very low measurement values or no analysis signal	■ Measuring gas connection incorrect	■ Check the measuring gas connection
	■ Gas outlet flow impeded	■ Check the "sample out" connection for unimpeded flow
	■ Oxygen supply interrupted	■ Check oxygen connection, reconnect as necessary
	■ Converter worn out/old	■ Inform customer service department
	■ Ozone generator faulty	■ Replace ozone generator, see (→ "Replacing the ozone generator" 141)
	■ Device leaking or defective	■ Inform customer service department
Results too high	■ High halogen contents disrupting TN determination	■ Dilute samples, if possible

Error	Possible cause	Remedy
Low results	■ Incomplete NO_x formation due to excessive nitrogen content ■ Nitrogen compounds cannot be completely converted to NO_x due to their structure (peptides, proteins, multiple N-N compounds such as azo dyes, polycondensed N compounds such as morpholine)	■ Reduce sample size/ amount ■ Dilute sample ■ Select an analysis method with O_2+ parameter mode
	■ Metallic cations in the sample lead to nitrogen salt formation	■ Dilute samples, if possible

13.9 Device faults on the chlorine detector

Error	Possible cause	Remedy
Drift out of range (displayed in the **Status analyzer** window	Drift > 100	■ Replace electrolyte
	Drift < -15	■ Check electrodes for wear, replace as necessary

13.10 Analytical problems during AOX, EOX and TX determination

Error	Possible cause	Remedy
Low results	■ Incomplete HX formation due too excessive halogen contents, due to excessive inorganically bound halogen compounds or due to metal ions working as catalysts (X_2 formation). NOTICE! The analysis system can become damaged, e.g., by chlorine.	■ Reduce sample size/ amount ■ Dilute sample
	■ Metallic cations in the sample lead to halogen salt formation	■ Dilute samples, if possible
	■ Sample unsuitable for vertical operation mode (droplet formation)	■ Use the horizontal operation mode
	■ In vertical operation mode: Quartz wool not present or at the wrong position in the combustion tube	■ Check the position of the quartz wool, adjust as necessary
	■ Sample evaporates before dosing	■ Use a cooled sampler.

Error	Possible cause	Remedy
	■ Sample too viscous too be drawn in without air pockets	■ Use the horizontal operation mode and dilute the sample, or dose directly as a solid.
	■ Inhomogeneous sample or sample containing particles	■ Homogenize sample
Results too high	■ High sulfur and nitrogen content disrupting determination	■ Dilute samples, if possible

13.11 Device faults on the S module 5100 basic and S module 5100 MPO

Error	Possible cause	Remedy
LED in front panel flashing	■ Run-in period not yet completed	■ Wait for the completion time for the run-in period of approx 30 min to elapse.
LED flashing after run-in time has elapsed	■ UV lamp defective	■ Check in **System** | **Component test** if a defective lamp is displayed ■ Replace lamp as necessary (→ "Replacing the UV lamp" 🖹 150) ■ If no defect is displayed, inform the customer service department
Detector sensitivity too low	■ Service life of the UV lamp expired	■ Replace lamp
Smell of ozone (only for sulfur detectors with MPO option)	■ Absorber on rear of module worn out or incorrectly connected	■ Check connection and replace absorber as necessary
	■ Gas hose on the absorber leaky or loose	■ Check hoses ■ Search for leak as necessary with indicator paper

13.12 Analytical problems during TS determination

These affect: Detection with the S module 5100 basic and the S module 5100 MPO

Error	Possible cause	Remedy
Scattering measurements	■ UV lamp defective	■ Replace lamp, see (→ "Replacing the UV lamp" 150)
	■ Sample unsuitable for vertical operation mode (droplet formation)	■ Use the horizontal operation mode
	■ In vertical operation mode: Quartz wool not present or at the wrong position in the combustion tube	■ Check the position of the quartz wool, adjust as necessary
	■ Sample evaporates before dosing	■ Use a cooled sampler.
	■ Sample too viscous too be drawn in without air pockets	■ Use the horizontal operation mode and dilute the sample, or dose directly as a solid.
	■ Inhomogeneous sample or sample containing particles	■ Homogenize sample
Low results	■ Incomplete SO_2 formation due to excessive sulfur content	■ Reduce sample size/ amount ■ Dilute sample
	■ Metallic cations in the sample lead to sulfur salt formation	■ Dilute samples, if possible
Results too high	■ Excessive halogen and nitrogen contents disrupt TS determination	■ Nitrogen: Use MPO technology ■ Dilute samples, if possible
	■ Incomplete sample incineration (pyrolysis products)	■ Use a suitable combustion mode or sampler, clean device before continuing work

13.13 Device faults on the coulometric sulfur detector

Error	Possible cause	Remedy
Stirring not working	■ Module not switched on	■ Switch on the module
	■ No stirrer rod in the measuring cell	■ Insert stirrer rod in the measuring cell
	■ Stirrer rod faulty	■ Replace stirrer rod
	■ Magnetic stirrer faulty	■ Inform customer service department

13.14 Analytical problems during coulometric TS determination

Error	Possible cause	Remedy
Scattering measurements	■ Sample unsuitable for vertical operation mode (droplet formation)	■ Use the horizontal operation mode
	■ In vertical operation mode: Quartz wool not present or at the wrong position in the combustion tube	■ Check the position of the quartz wool, adjust as necessary
	■ Sample evaporates before dosing	■ Use a cooled sampler.
	■ Sample too viscous too be drawn in without air pockets	■ Use the horizontal operation mode and dilute the sample, or dose directly as a solid.
	■ Inhomogeneous sample or sample containing particles	■ Homogenize sample
False measurement values	■ Stirrer function faulty	■ To remedy, see (→ "Device faults on the coulometric sulfur detector" 🖹 116)
	■ Incorrect electrolyte	■ Create electrolyte, see (→ "Preparing the measuring cell" 🖹 80)
	■ Filling level in the measuring cell too high or low	■ Fill the measuring cell of to the height of the port for manual measurement
	■ Electrolyte used up	■ Replace electrolyte
	■ Electrodes incorrectly connected or faulty	■ Check electrode connections or replace electrodes
Low results No analysis signal	■ Measurement gas transfer to the measuring cell interrupted	■ Check hose connections
	■ Glass or hose components are wet	■ Dry glass/hose components
Results too high	■ Excessive nitrogen content and heavy metal ions disrupt TS determination	■ Check the NOx and HX absorbers, refill as necessary ■ Replace the electrolyte solution daily to prevent formation of disruptive ions

13.15 Device faults on the carbon detector

Error	Possible cause	Remedy
Analog values exceeding values range (display in the **Status analyzer** window)	■ Analog values outside of operating range	■ Check the gas connection to the basic module ■ Check the gas quality ■ Check the analog values by performing a component test: **System** menu**Component test**
	■ NDIR detector faulty	■ Inform customer service department

13.16 Analytical problems during TC, EC/OC determination

Error	Possible cause	Remedy
Scattering measurements	■ Inhomogeneous sample matrix or sample matrix containing particles	■ Warm up cold samples ■ Homogenize sample before analysis
	■ NDIR baseline drift ■ Unfavorable integration criteria: Integration canceled to early	■ Check settings ■ Increase the maximum integration time
	■ Sample unsuitable for vertical operation mode (droplet formation)	■ Use the horizontal operation mode
	■ In vertical operation mode: Quartz wool not present or at the wrong position in the combustion tube	■ Check the position of the quartz wool, adjust as necessary
	■ Sample evaporates before dosing	■ Use a cooled sampler.
	■ Sample too viscous too be drawn in without air pockets	■ Use the horizontal operation mode and dilute the sample, or dose directly as a solid.
	■ Inhomogeneous sample or sample containing particles	■ Homogenize sample
Low results	■ CO_2 concentration outside of the operating range of the NDIR detector	■ Reduce sample volume/ amount ■ Dilute samples
	■ Inlet gas flow set too low in the method (EC/ OC methods only)	■ Adjust method parameters
Results too high	■ Inlet gas flow set too low in the method (EC/ OC methods only)	■ Adjust method parameters
No analysis signal	■ NDIR detector faulty	■ Inform customer service department

13.17 Device faults on the TOC detector

Error	Possible cause	Remedy
Analog values exceeding values range (display in the **Status analyzer** window)	■ Analog values outside of operating range	■ Check the gas connection to the basic module ■ Check the gas quality ■ Check the analog values by performing a component test: **System** menu**Component test**
	■ NDIR detector faulty	■ Inform customer service department
Leaking condensate pump	■ Leaking hose connections ■ Pump hose faulty	■ Replace the pump hose
Sample is not drawn up without air bubbles	■ Leaking syringe	■ Check the dosing syringe ■ If leaking, replace syringe
	■ Clogged cannula ■ Wrong cannula used	■ Remove the cannula and clean it in an ultrasound bath Replace cannula as necessary
	■ Dosing syringe contaminated by grease/oil	■ Clean the dosing syringe with the following solutions: Tenside solution, 30 min acting time NaOH (0.1 mol/l), 10 min acting time HCl (0.1 mol/l), 10 min acting time ■ Rinse the syringe thoroughly with ultrapure water after each cleaning step
Water traps clogged	■ Service life expired (> 6 months)	■ Replace the water traps ■ Do not analyze any samples that form aerosols ■ Only acidify samples with hydrochloric acid
	■ Water trap capacity exhausted due to excessive aerosol formation	

13.18 Analytical problems during TC, EC/OC, TOC, NPOC and TIC determination

TC and EC/OC determination in organic liquids, in solids and in gases:

Error	Possible cause	Remedy
Scattering measurements	■ Inhomogeneous sample matrix or sample matrix containing particles	■ Warm up cold samples ■ Homogenize sample before analysis
	■ NDIR baseline drift ■ Unfavorable integration criteria: Integration canceled to early	■ Check settings ■ Increase the maximum integration time
	■ Sample unsuitable for vertical operation mode (droplet formation)	■ Use the horizontal operation mode
	■ In vertical operation mode: Quartz wool not present or at the wrong position in the combustion tube	■ Check the position of the quartz wool, adjust as necessary
	■ Sample evaporates before dosing	■ Use a cooled sampler.
	■ Sample too viscous too be drawn in without air pockets	■ Use the horizontal operation mode and dilute the sample, or dose directly as a solid.
	■ Inhomogeneous sample or sample containing particles	■ Homogenize sample
Low results	■ CO_2 concentration outside of the operating range of the NDIR detector	■ Reduce sample volume/ amount ■ Dilute samples
	■ Inlet gas flow set too low in the method (EC/ OC methods only)	■ Adjust method parameters
Results too high	■ Inlet gas flow set too low in the method (EC/ OC methods only)	■ Adjust method parameters
No analysis signal	■ NDIR detector faulty	■ Inform customer service department

TC, TOC, NPOC and TIC determination in water analysis:

Error	Possible cause	Remedy
Scattering measurements	■ Filling of combustion tube depleted	■ Replace catalyst, see (→ "Replacing the catalyst in the TOC combustion tube" 🖹 162)
	■ Dosing faulty	■ Check dosing ■ For manual dosing: Check syringe volume
	■ Cannula damaged or clogged	■ Replace cannula or remove clogging with a cleaning stylet ■ Filter samples containing particles before analysis
	■ Inhomogeneous samples	■ Inhomogeneous water samples with a high organic content, for example oils, must be homogenized and may only be analyzed in horizontal operation mode (with ABD)
	■ Faulty, leaky septum	■ Check septum, replace as necessary ■ Only use cannula with ID 0.35 mm for 250/500 µl special syringes
	■ Sample contamination with ambient air contents	■ Check ambient conditions and remedy the source of the fault
	■ NDIR baseline drift ■ Unsuitable integration criteria: Integration canceled too early or excessive duration (baseline noise integrated)	■ Check the gas supply and gas quality ■ Adjust the maximum integration time or the start and stop criteria
Low results	■ Catalyst depleted	■ Exchange the catalyst
	■ System is leaking	■ Check the sluice for leaks ■ Replace septum
	■ Incorrect injection volume	■ For manual sampling: introduce the sample volume set in the method
	■ Phosphoric acid in the TIC reactor depleted	■ Regenerate the TIC reactor, see (→ "Regenerating the TIC reactor" 🖹 159)
	■ Septum faulty	■ Replace septum

14 Maintenance and care

14.1 Overview of maintenance work

Basic module

Maintenance interval	Measure
Daily and after maintenance	Check gas flow
	Check system tightness
Weekly	Clean and care of the analyzer
	Check all hose connection for proper fit; replace loose connections
Monthly	Check fastening screws for proper fit; tighten loose screw connections
	Check combustion tube for damage
	Check FAST connectors at the combustion tube for proper fit, cracks of damage; replace damaged FAST connectors
Quarterly	Auto-protection valve assembly: Check filters
Annually	Gas box: Check check valve and inlet filter
As necessary	Replace combustion tube if it shows cracks, devitrification or other damage
	Replace check valve and inlet filter in the gas box if components are clogged/damaged and block the gas flow
	Check the correct position of the quartz wool used in vertical operation mode in the combustion tube (for example after replacing the septum or a FAST connector on the combustion tube)

N module 5100

Maintenance interval	Maintenance task
Weekly	Clean the module from the outside
	Check hoses for cracks, replace as necessary
	Check hose connections for tight fit
Annually	Replace the chemical ozone decomposer
	Replace the ozone generator (recommend during annual routine maintenance)
	Replace the NO converter tube (by customer service department, recommended during annual routine maintenance)
As necessary	Replace the absorber if the baseline is too high

S module 5100 basic, S module 5100 MPO

Maintenance interval	Maintenance task
Weekly	Clean the module from the outside
	Check all screw connections for tight fit
Annually	Replace the absorber (S module 5100 MPO only)
As necessary	Replace the UV lamp

Cl module 5100

Maintenance interval	Maintenance task
Daily	Replace sulfuric acid daily or when empty
	Measuring cells "sensitive" and "high concentration": Change the electrolyte, wipe out measuring cell after each electrolyte replacement
	Measuring cell "high sensitive" : ■ Refill electrolyte, Electrolyte replacement only if crystalline deposits form, if the electrolyte has become opaque or the measurement sensitivity is reduced ■ Clean and dry the sulfuric acid container and safety attachment including connectors and gas inlet hose
Weekly	Clean the chlorine module
	Check all screw connections for tight fit
	Measuring cell "high sensitive": ■ Check the bridge electrolyte fill level of the reference electrode, refill as necessary ■ Replace bridge electrolyte if the fill level has dropped to 2 cm below the opening or after multiple refills
	Clean and dry the sulfuric acid container and safety attachment including connectors and gas inlet hose (when using the "sensitive" and "high concentration" measuring cells)
	Clean measuring cell
	Clean the measuring gas hose/gas transfer line including connector with distilled water and dry via blowing out with an inert gas
Monthly	Check hoses for cracks and proper fit, replace as necessary
	Check the ferrules in the Swagelok connectors (PTFE) for damage, replace as necessary

S module 5100 coulometric

Maintenance interval	Maintenance task
Daily	Replace electrolyte
Weekly	Clean the module from the outside
	Check all screw connections for tight fit
	Check the filling of the NOx and HX absorbers, replace as necessary
	Clean measuring cell
Quarterly	Check measuring cell for cracks and damage, replace as necessary

C module 5100

Maintenance interval	Maintenance task
Weekly	Clean the module from the outside
	Check hoses for cracks, replace as necessary
	Check hose connections for tight fit

TOC module 5100

Maintenance interval	Maintenance task
Daily	Check gas flow
	Check copper wool in the halogen trap for discoloration
	Regenerate the TIC reactor
Weekly	Clean the module from the outside
	Check hoses for cracks, replace as necessary
	Check hose connections for tight fit
Quarterly	Check the TOC combustion tube for cracks and damage
	Check the TIC reactor for cracks and damage.
	Check the condensation coil for cracks and damage
	Check the condensate pump for leaks
	Check the dosing syringe for leaks
Every six months	Replace water traps, earlier if necessary
Annually	Replace catalyst in the TOC combustion tube, earlier if instructed in the software
	Clean the TOC combustion tube
	Clean the condensation coil
	Replace the pump hose of the condensate pump
	Clean the dosing syringe
As necessary	Replace the filling of the halogen trap as soon as half of the copper wool has changed color
	Replace the septum on the TOC combustion tube if the system has any leaks
	Replace the septum on the TIC reactor if the system has any leaks

14.2 Maintenance of the multi-purpose combustion tube

CAUTION

Risk of injury from falling components

The user can be injured if the combustion tube falls down during maintenance.

- Exercise extreme caution when performing maintenance on the combustion tube.

14.2.1 Removing the combustion tube

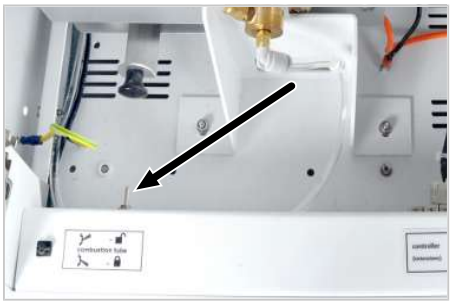
CAUTION

There is a risk of burns at the combustion furnace and combustion tube!

- Only perform removal when cold. Allow the device to cool sufficiently.
- Wear the heat-proof gloves included in the delivery when handling hot components. These gloves are suitable for temperatures of up to 200 °C.

Maintenance of the combustion tube is always performed when the furnace is is the vertical installation position. Remove the combustion tube as follows:

- Exit multiWin.
- Switch off the basic module via the power switch and shut off the gas supply.
- Remove the upper cover of the basic module.



- Open the device door. Flip the toggle switch for the pneumatic seal up.
  ✓ The auto-protection valve assembly is opened.

- Horizontal operation mode with ABD:
  Unscrew the coupling at the connection between the combustion tube and the ABD. Slide the ABD back slightly. Rotate the furnace to the vertical position.

- Remove hose 3 and hose 4 from the FAST connectors on the combustion tube.
- Horizontal operating mode:
  Carefully grip the flame sensor (FS) by the blue ring and pull it off the combustion tube.
  The connection to the combustion tube is very fragile!
- Carefully remove the combustion tube from the furnace.
- Check the combustion tube for crystallization, cracks and burst spots.
- If the vertical operation mode is in use: Check the condition and position of the quartz wool plug

14.2.2 Cleaning the combustion tube

⇨ Remove the combustion tube from the combustion furnace (→ "Removing the combustion tube" 🖹 125).

- Remove the screw cap and septum, if present. Remove the 2 angled FAST connectors from the combustion tube.
- For nitrogen and sulfur determination in vertical operation mode: Remove the quartz wool plug from the combustion tube with a long hook.
  Wear protective clothing when replacing the quartz wool (labcoat, protective gloves and glasses) Wear a respiratory mask or work underneath an exhaust vent because quartz wool dust irritates the respiratory tracts.
- Clean the inside of the combustion tube with a suitable solvent and a cotton swab or bottle brush. Rinse with distilled water if the solvent is water-soluble. Otherwise, rinse with ethanol.
- Dry the combustion tube (e.g., by blowing it through with an inert gas).
- Deposits from incomplete incineration, e.g., soot or solid pyrolysis residue, can also be removed by firing the combustion tube in a muffle furnace at 750–900 °C or with a suitable burner flame, e.g., propane.
- For nitrogen and sulfur determination in vertical operation mode: Insert a new quartz wool plug in the combustion tube (→ "Inserting the quartz wool plug" 🖹 126).

✓ The combustion tube is clean and can be reinserted.

14.2.3 Inserting the quartz wool plug

For nitrogen and sulfur determination in vertical operation mode: Insert a quartz wool plug in the combustion tube.

Missing quartz wool leads to sooting of the analysis system. Sample with high salt content form ash and solid oxides during incineration that can build up in the quartz wool. The quartz wool must then be replaced. The quartz wool plug is not needed for operation in horizontal mode.

CAUTION

Skin and respiratory system irritation due to quartz wool

Quartz wool tends to form dust. Irritation can occur after breathing in or skin contact with this dust.

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

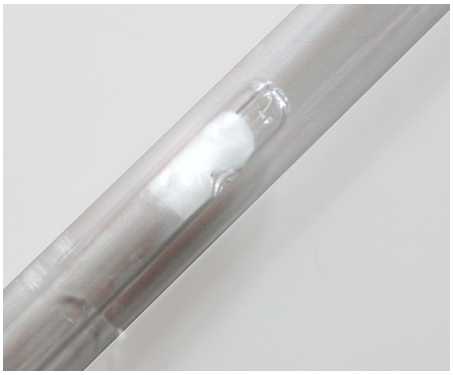
NOTICE

Risk of device damage

- Only use the pure quartz wool supplied by Analytik Jena GmbH+Co. KG. Contaminated quartz wool may damage the combustion tube and clog the filter.
- Ensure correct positioning of the quartz wool plug. When the plug is not in correct position the sample does not vaporize evenly.

- Remove the combustion tube from the combustion furnace as described.

- Roll a small amount of quartz wool into a loose plug approx. 1.5 to 2 cm long.



- Insert the quartz wool plug into the inner tube of the combustion tube with a clean glass rod.
- Push the quartz wool plug into the tube until the positioning mandrel is at the center of the plug.
  The plug must not seal the bottom slot in the inner tube.
  The plug should cover the entire cross section of the inner tube.
- After replacing the quartz wool: Clean the analysis system by performing at least 3 measurements with a pure solvent (e.g., isooctane, toluene, xylene).

14.2.4 Installing the combustion tube

WARNING

Risk of explosion and sooting due to improper connection of the gases to the combustion tube.

- The connections for argon and oxygen on the combustion tube must not be mixed up!

CAUTION

Risk of burns from hot components and possible damage to the seal of the auto-protection valve assembly.

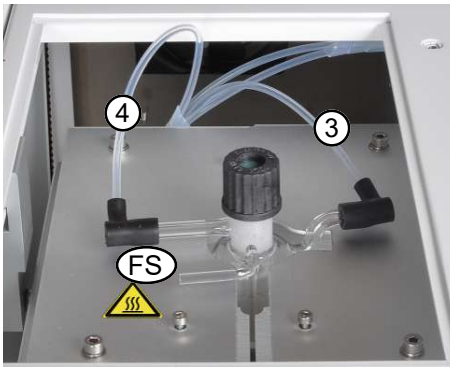
- Allow the combustion tube to cool before cleaning after incineration.
- Allow the combustion furnace to cool before inserting the combustion tube.

NOTICE

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

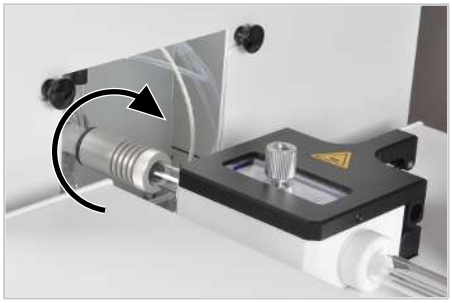
- Wear protective gloves when inserting the combustion tube and do not touch the tube with bare hands.
- Wipe the outside of the combustion tube off with ethanol and a paper towel before insertion.

- Place the combustion system in the vertical position.
- For nitrogen and sulfur determination in vertical operation mode: Ensure that the quartz wool plug has been inserted at the proper place in the combustion tube.
- Apply FAST connectors to the gas connections of the combustion tube.
  NOTICE! For angled FAST connectors: Do not push the combustion tube connections too far into the limbs of the FAST connectors. The gas flow may be impeded otherwise.
- Insert the combustion tube into the combustion furnace. The bent gas connection for hose 3 must be fit into the recesses on the furnace.

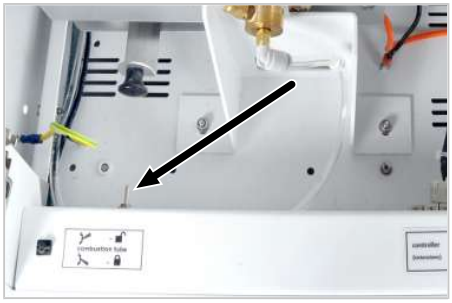


- Vertical operating mode:
  Screw a screw cap with septum onto the combustion tube.
  Press hoses 3 and 4 into the FAST connectors on the combustion tube.
- Re-insert the cover plate with hole into the top device opening.
  Connect the autosampler or auto-injector.

- For horizontal operating mode:
  Press hoses 3 and 4 into the FAST connectors on the combustion tube.
  Carefully press the flame sensor (FS) onto the combustion tube connection. The connection is very fragile!
  Move the combustion furnace into the horizontal position.



- Connecting the ABD:
  Check the sealing element in the coupling piece of the ABD for correct fit and insert new a sealing element into the coupling if necessary.
  ABD Fasten the ABD with the coupling piece to the combustion tube.
  Also see the "ABD user manual".



- Open the gas supply at the pressure reducer.
- Close the pneumatic seal at the auto-protection valve assembly. Flip the toggle switch down.
  ✓ The combustion tube is now sealed in the auto-protection valve assembly and ready for operation again.

14.3 Maintenance of the auto-protection valve assembly

CAUTION

Risk of injury from falling components

The user can be injured if the assembly falls down during maintenance.

- Exercise extreme caution during maintenance of the auto-protection valve assembly.

14.3.1 Installing/removing the auto-protection valve assembly

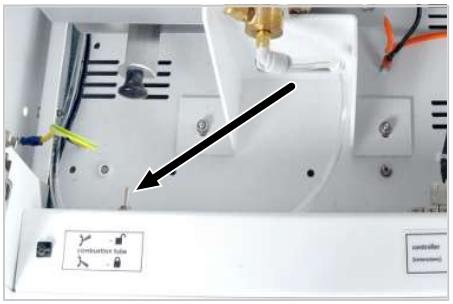
CAUTION

Risk of burns from the hot furnace and the gas transfer line.

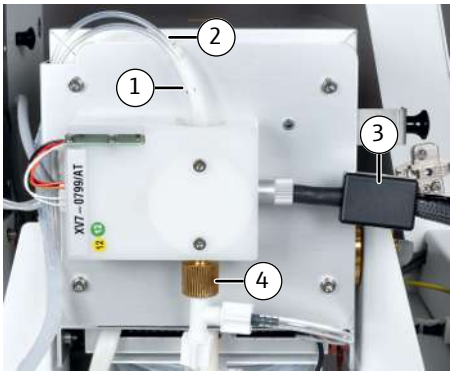
- Switch off the device and allow it to cool before performing maintenance.

Check the condition of the auto-protection valve assembly as follows:

For better clarity, the work steps are displayed with the side panels removed. However, for the installation and removal of the auto-protection valve assembly it is not necessary to remove the side panels.



- Exit the multiWin program, switch off the basic module via the power switch and shut off the gas supply.
- Tilt the combustion furnace into the horizontal position.
- Open the pneumatic seal at the auto-protection valve assembly. Flip the toggle switch up.
- Remove the combustion tube or pull it out some distance of the combustion furnace.

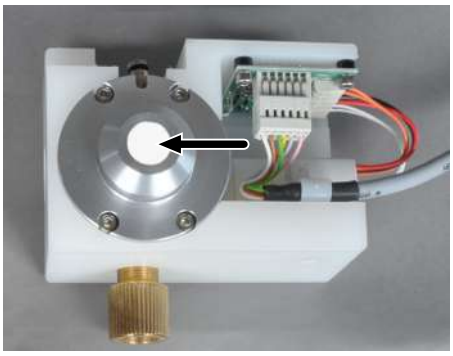


- Unscrew hose 8 (1) from the connection.
- Press the ring on the connector of hose 11 (2) down and pull the hose out of the connection.
- Depending on the configuration, detach the connection of the membrane dryer and the gas transfer line:
  - Unscrew the gas transfer line (3).
  - Loosen the knurled screw (4) on the membrane dryer connection slightly and pull the connection out toward the bottom.

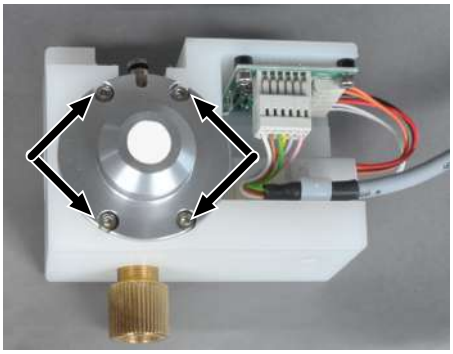
- Pull the electrical connection of the auto-protection valve assembly and, where applicable, the transfer line out of the connection.

- Hold the auto-protection valve assembly with the left hand and pull at the knob of the clamping mount with the right hand to open the lock. Remove the auto-protection valve assembly from the combustion furnace.
- The installation of the auto-protection valve assembly is in reverse order.

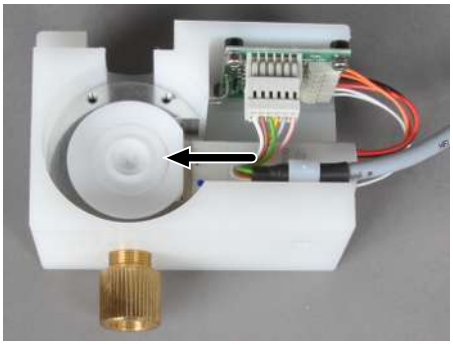
14.3.2 Check and replace the filter



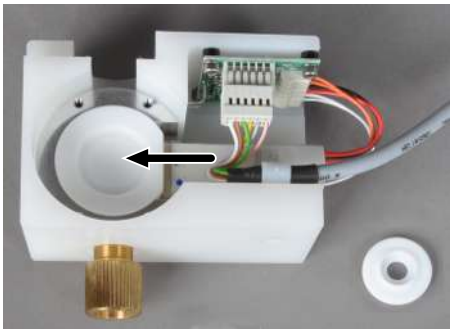
- Remove the auto-protection valve assembly.
- Check the filter for carbonization, soiling or cracks through visual inspection.
  - If the filter is OK, reinstall the auto-protection valve assembly.
  - If the filter must be replaced, continue to follow the instructions.



- Unscrew the 4 screws attaching the pneumatic seal to the auto-protection valve assembly.
- Remove the pneumatic seal from the auto-protection valve assembly.

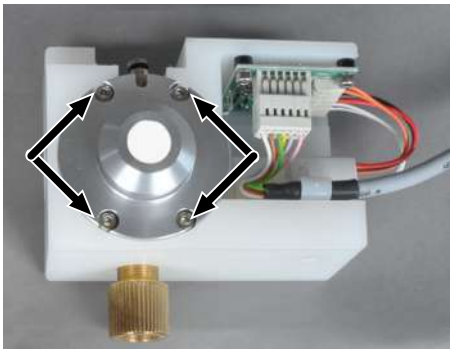


- Remove the intermediate ring.



- Remove the worn filter and insert a new filter.
- Reassemble the auto-protection valve assembly again in reverse order.
  - ✓ The auto-protection valve assembly is ready for operation again.

14.3.3 Replacing the pneumatic seal

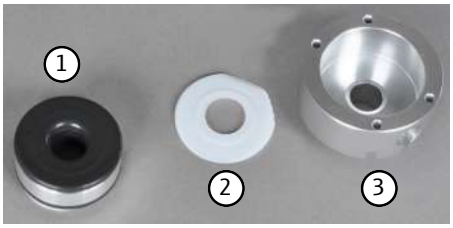


- Remove the auto-protection valve assembly.
- Unscrew the 4 screws attaching the pneumatic seal to the auto-protection valve assembly.

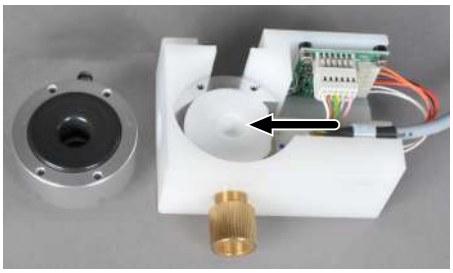
- Remove the housing with the pneumatic seal from the auto-protection valve assembly.
- Unscrew the connection bushing for hose 11 from the seal housing.

- Remove the pneumatic seal from the housing.
- Remove the PTFE disks from both sides of the seal.

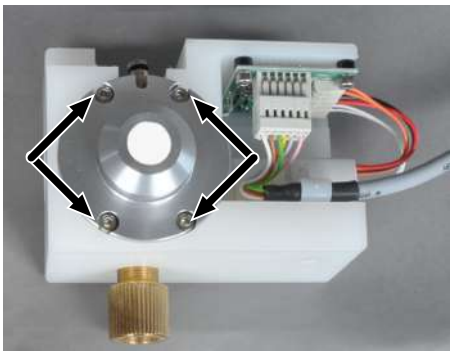
- Carefully separate the special seal from the ring.
- Insert a new special seal into the ring.



- Place the PTFE disk (2) in the housing (3).
- Place the seal (1) in the housing. The hole in the ring must align with the hole in the housing.
- Screw in the connection socket for hose 8.



- Place the second PTFE disk onto the intermediate ring above the filter.



- Place the pneumatic seal onto the auto-protection valve assembly and attach it with 4 screws.
  - ✓ The auto-protection valve assembly is ready for operation again.

14.4 Replacing the membrane dryer

CAUTION

Risk of burns from the hot furnace

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

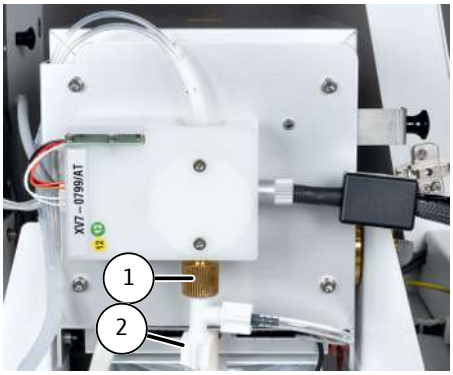
NOTICE

Risk of damage due to pinching or twisting

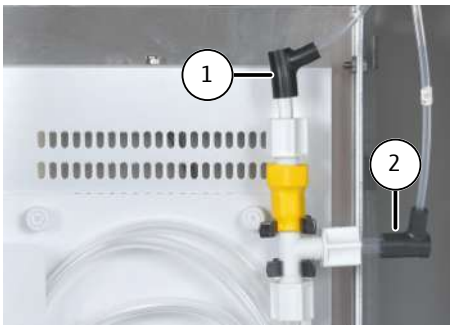
The sensitive membrane for water vapor exchange in the membrane dryer can be damaged by squeezing or twisting.

- Do not pinch the membrane when installing the new membrane dryer.
- Do not twist the sensitive connections.

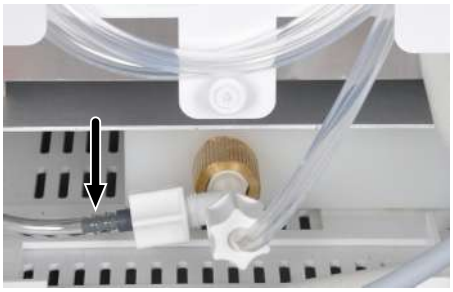
For better clarity, some work steps are displayed with the side panels removed. However, for the installation and removal of the membrane dryer it is not necessary to remove the side panels.



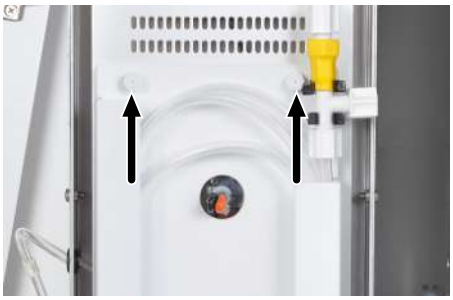
- Move the combustion furnace into the horizontal position.
- Detach the connection of the membrane dryer at the auto-protection valve assembly. Slightly loosen the knurled screw (1) and pull the connection (2) out toward the bottom.



- Move the combustion furnace into the vertical position.
- Remove hose 5 (1) and hose 12 (2).



- Remove hose 13 (see arrow).



- Unscrew the 3 knurled screws and remove the holder.
  The membrane dryer is fastened to the furnace with 2 knurled screws on the top side (see arrows) and 1 knurled screw on the bottom.
- Remove the old membrane heater from the mount.

- Carefully place the new membrane dryer in 2 strap loops, insert and clamp it in the holder.
- The gas connection at the top end must point to the right and the gas connection at the bottom end to the left.
  NOTICE! The connections must not be pinched or twisted.
- Reinstall the holder with the new membrane dryer in reverse order.

✓ The membrane dryer is installed and ready for operation.

14.5 Replacing the hose connections

Check the hose connections regularly for leaks. Remove and replace faulty hoses and hose connections. Check the system for leaks after maintenance (→ "Checking the system for leaks" 137).

When replacing Fingertight screw connections, observe the following:

- Only use straight cut, round and unpinched hose ends for the connection.
- Slide the conical nipple with the conical side towards the banjo bolt onto the hose.
- The conical nipple and hose end must be flush.

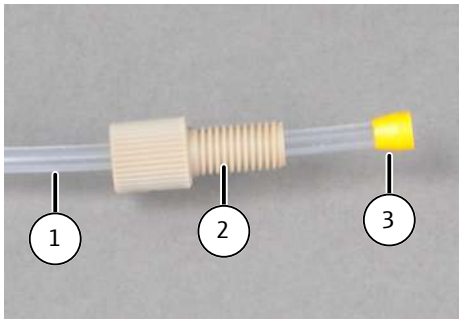


Fig. 66 Replacing the Fingertight connections

1 Hose
2 Banjo bolt
3 Conical nipple

14.6 Replacing the septum on the injection port

In vertical operation mode, the septum on the injection port of the combustion tube must be replaced if it is worn out, as this leads to leaks in the system.

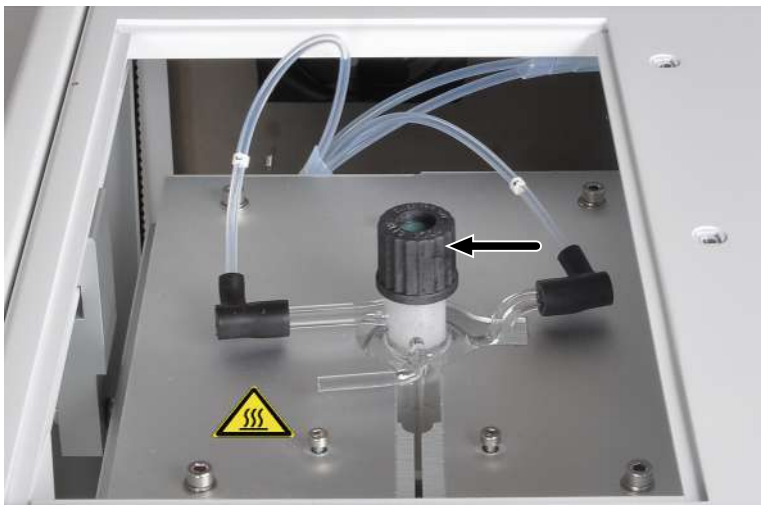


Fig. 67 Replacing the septum on the injection port of the combustion tube

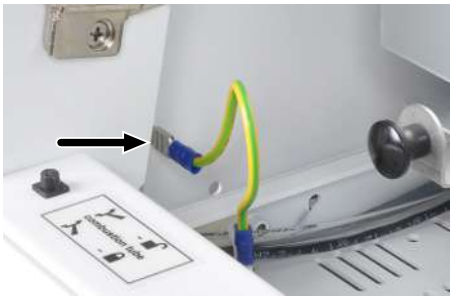
- Open the device door. Flip the toggle switch of the pneumatic seal up to open the auto-protection valve assembly.
- Remove the cover from the top of the basic module.
- Unscrew the screw cap of the combustion tube.
- Check that the quartz wool plug is still in the proper position in the inner tube of the combustion tube.

- Insert the septum and screw the screw cap back onto the combustion tube.
  ✓ Replacing the septum on the combustion tube is now complete.

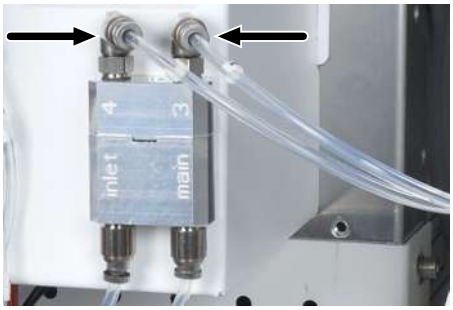
14.7 Replacing the check valves and particle filters

14.7.1 Replacing the check valves on the gas box

The check valves must be replaced if the gas flow can no longer be set to the target value (observe the message in the software) and possible leaks in the system have been excluded. The check valves are located in the valve block on the gas box on the left side of the device.



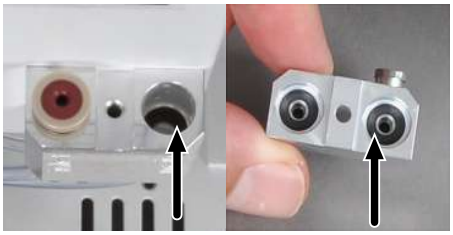
- Switch off the basic module and disconnect the power plug from the power outlet.
- Switch off the gas supply at the shut-off valve.
- Remove the protective ground conductor on the left side panel. Loosen the 4 screws on the left side panel and lift off the side panel.



- Pull hoses 3 and 4 out of their connections on the valve block (see arrows).

- Unscrew the screw on the valve block with a 2.5 mm hexagon socket wrench.

- Remove the top part of the valve block and take the check valves out of the "main" and "inlet" connections.

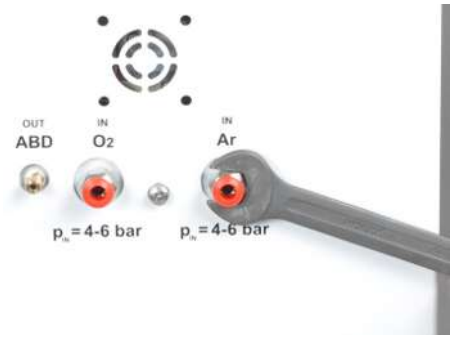


- Replace the sealing rings of the check valves in the upper and lower part of the valve block.
- Insert new check valves.
- Assemble the valve block and screw on the top part.
- Connect hose 3 to the "main" connection and hose 4 to the "inlet" connection.
- Connect the protective ground conductor to the side panel and close the side panel.
- Open the gas supply at the shut-off valve.
- Connect the power cable on the basic module and switch on the module via the power switch.

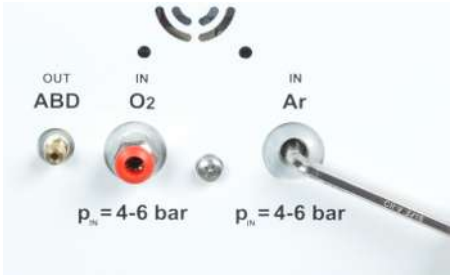
  ✓ The basic module is now ready for operation again.

14.7.2 Replacing the particle filters in the gas inlets

The "Ar" and "O2" gas inlets on the rear of the basic module are equipped with particle filters. The particle filters and check valves must be replaced if the process gases can no longer be set to their target values (observe the message in the software) and possible leaks in the system have been excluded.



- Switch off the basic module and disconnect the power plug from the power outlet.
- Switch off the gas supply at the shut-off valve.
- When using the ABD: Disconnect the ABD from the basic module and move the sampling module slightly to access the rear of the device.
- Pull the gas hose out of its connection on the rear of the basic module. To do so, press down the red ring and pull the gas hose out of the connection.
- Unscrew the gas connections with a 13 mm open-ended wrench.



- Unscrew the particle filters inside using a 5 mm hexagon socket wrench.

- Insert and tighten the new particle filter.
- Screw in the gas connections and tighten them with an open-ended wrench. Connect the gas hoses.
- Reconnect the sampling module, if necessary.
- Switch on the gas supply.
- Connect the power cable to the basic module and switch on the module via the power switch.

  ✓ The basic module is now ready for operation again.

14.8 Checking the system for leaks

- Switch on the basic module and the system components.
- Open the gas supply.
- Start the multiWin program.
- Activate a method.
  - ✓ The current gas flows are displayed in the **Status analyzer** window.

Faulty gas inlet flows are marked in red in the **Status analyzer** window.

MFC 1	200 ml/min	Oxygen (primary oxygen), hose 3, value cannot be changed in the method
MFC 2	0 ml/min (idle state)	Oxygen for post-combustion phase, hose 4, gas inlet on the combustion tube, value is set in the method
MFC 3	100 to 200 ml/min (example)	Pyrolysis gas (argon), hose 4, gas inlet on the combustion tube, value is set in the method

14.8.1 System tightness for N/S/C methods

The system tightness for N/S/C methods is monitored automatically. If the system is leaking, the **Gas leak** message will appear in the **Status analyzer** window and the MFC 1 display is marked in red. Starting a measurement is not possible.

14.8.2 System tightness for Cl methods

WARNING

Risk of chemical burns

Concentrated sulfuric acid is used in the detection module as a drying agent. The concentrated acid can lead to severe chemical burns.

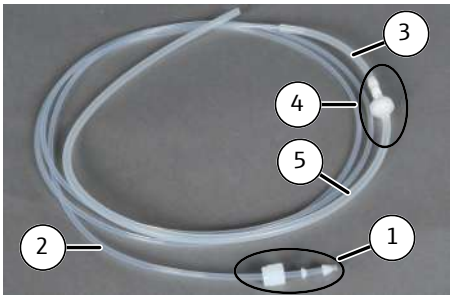
- Wear protective clothing when working with these hazardous substances.
- Completely empty the sulfuric acid container prior to the system tightness check.
- Observe all notes and specifications in the safety data sheet.

NOTICE

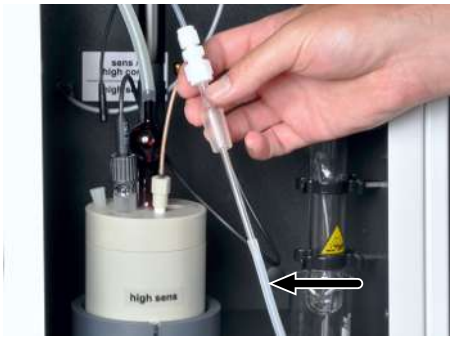
Risk of destruction of the internal MFM due to corrosive gas

- To check the system tightness, only use the flow monitoring set included in the scope of delivery.

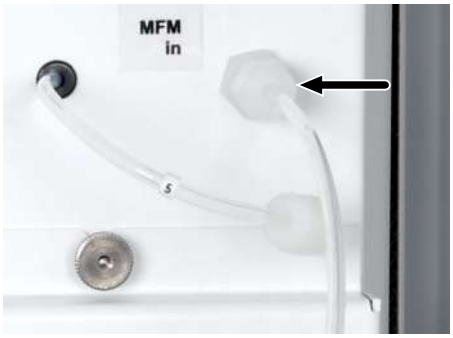
The check of the system tightness is not performed automatically, but instead manually using the hose set (flow monitoring set) included in the scope of delivery.



- Assemble the flow monitoring set in the following order:
  - Thread the screw cap, the seal and the sealing cone (1) onto the thin hose (2).
  - Connect the hose (2) with the other hose (3).
  - Attach the water trap and the adapter (4) to the hose (3).
  - Attach the hose (5) to the adapter.

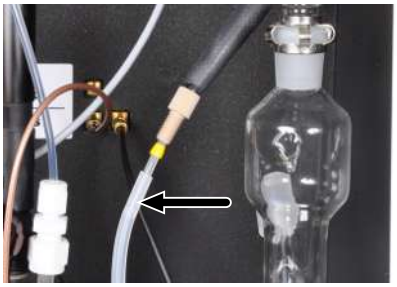


- First, completely empty the sulfuric acid from the sulfuric acid container of the CI module 5100 (→ "Replacing the sulfuric acid and cleaning the sulfuric acid container." 145). Then clean, dry and re-install the container and all adjacent components (safety attachment, gas transfer hose, connector).
- Measuring cell "high sensitive" (image):
  - Remove the gas inlet hose from the measuring cell. Remove the electrolyte from the gas inlet hose. Clean and dry the outside and inside of the tube and the connector.
  - Connect the gas inlet hose to hose 5 of the flow monitoring set.
- Measuring cells "sensitive" and "high concentration":
  - Disconnect hose 20 from the combined electrode and connect it to hose 5 of the flow monitoring set.



- Remove the "MFM in" screw connection on the cover of the control electronics in the basic module and connect the flow monitoring set (hose 2) (see arrow).
- Read the current gas flow in the **Component test** | **Flow** System | **Component test** menu item) window.
  The target flow is the sum total of the measured inlet flows (main + inlet + argon bypass). For methods using gas samplers, the auxiliary gas flow of the gas sampler must be added.
- If the displayed flow differs from the target flow by more than ± 15 ml/min, search for and remedy the possible causes. Contact the customer service department if this is not successful.
- After the flow measurements, remove the set and reconnect hose 5 to the MFM inlet to ensure a complete measuring gas path for N/S/C methods.
- Refill the sulfuric acid container with sulfuric acid.

Alternatively, the system tightness can also be checked at the transfer line. In this case, it is not necessary to drain the sulfuric acid, but the check does not cover the entire gas path. As leaks usually occur on the basic module, this check is a simple and quick alternative.



- Open the front doors of the basic module and the CI module 5100.
- Unscrew the heated transfer line from the connector of the sulfuric acid container.
- Connect the Fingertight connector of the transfer line to the hose of the flow monitoring set.
- Then proceed as described before.

14.8.3 System tightness for TOC methods

System tightness for the gas path from the inlet of the basic module to the outlet of the TOC module 5100 is not regulated automatically. Use the hose set included in the scope of delivery and proceed as follows:

⇨ The basic module and the detection module are switched on and connected.

⇨ The carrier gas supply is open.

⇨ The multiWin control and analysis software has been started.

⇨ A method for TOC determination is active (see software manual).

- Assemble the flow monitoring set (→ "System tightness for Cl methods" 🖹 137).
- Connect hose 5 of the flow monitoring set to the "sample out" outlet on the rear of the detection module.
- Open the doors of the basic module.
- If necessary, disconnect the connection on the "MFM in" connection. The connection is located on the cover plate of the control electronics on the right in the basic module (→ "System tightness for Cl methods" 🖹 137).
- Connect the other end of the hose set with the "MFM in" inlet on the basic module.
- Read the current gas flow in the **System** | **Component test** menu under **Device** | **Control flow**.
- If the displayed flow differs from the target flow by more than ± 5 ml/min, search for and remedy the possible causes. Contact the customer service department if this is not successful.

  ✓ The target flow is displayed in the **Status analyzer** window:

	Target	Description
MFC 1	200 ml/min	Primary oxygen (hose 3) in the basic module, value cannot be changed in the method
MFC 2	0 ml/min	MFC 2 and MFC 3 are in standby during TOC operation.
MFC 3		

14.9 Removing and installing the combustion furnace

WARNING

Risk of electrical shock

- Before installing the combustion furnace, switch off the basic device via the power switch and disconnect the power plug from the power outlet!

CAUTION

Risk of burns from the hot furnace

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

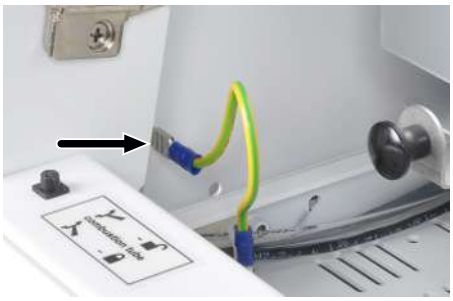
CAUTION

Risk of injury from falling components

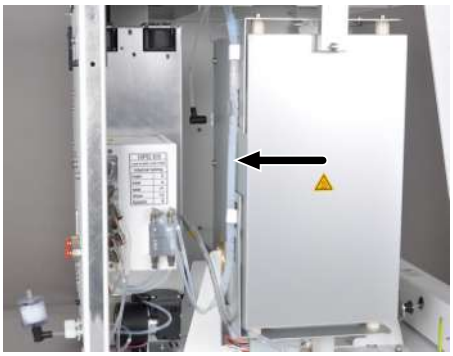
The user can be injured if the combustion furnace falls down during installation/removal.

- Exercise extreme caution when installing/removing the combustion furnace.

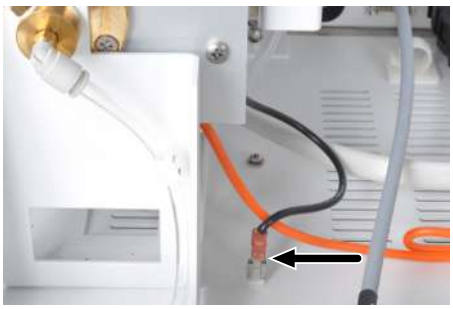
The combustion furnace must be removed for transport.



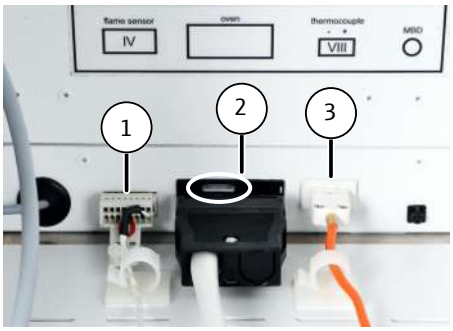
- Remove the combustion tube (→ "Maintenance of the multi-purpose combustion tube" 🖹 124). Leave the furnace in the vertical position after this.
- Exit the multiWin program.
- Switch off the basic module via the device switch and disconnect the power plug from the socket. Switch off the gas supply.
- Remove the top cover and the doors from the module.
- Remove the left side panel: Disconnect the protective ground conductor. Loosen the screws at the left side panel. Lift the side panel off and store it safely.



- Remove the hoses from their holders on the combustion furnace (see arrow).

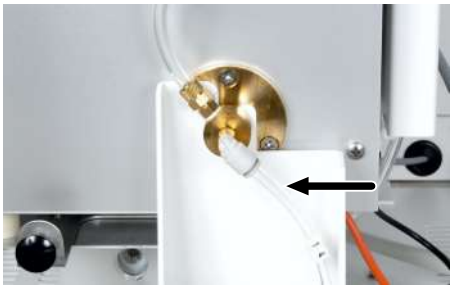


- Remove the protective grounding from the base plate of the combustion furnace.



- Remove the three plug-in connectors from their sockets:
  - Flame sensor (1)
  - Electrical connection of the combustion furnace (2). Press the gray lever up slightly when doing this.
  - Thermoelement (3) with colored cable

- Move the combustion furnace into the horizontal position.
- Remove the auto-protection valve assembly from the combustion furnace (→ "Maintenance of the auto-protection valve assembly" 129).



- Detach hose 14 (see arrow). Press the ring at the plug connector into the connector and pull off the hose.
- If necessary, remove the connections on the membrane dryer (for S/N/C methods).
- Carefully lift the combustion furnace of the basic module.
- The installation of the combustion furnace container is performed in the reverse order.

14.10 Maintenance of the N module 5100 nitrogen detector

14.10.1 Replacing the ozone generator

WARNING

Risk of electric shock

High voltages are present in the interior of the device, which can lead to electric shock if contacted.

- Before opening: Switch off the device via the power switch.
- Disconnect the power cable from the socket.

CAUTION

Risk of burns from the thermal ozone decomposer!

- Only perform maintenance in the inside of the device when cold/allow the device to cool sufficiently.

CAUTION

Risk of respiratory problems due to leaking ozone

If the gas hoses are not properly connected to the ozone generator, ozone can leak out of the detection module.

- Ensure proper hose connection.
- Check that the gas connections are sealed with indicator paper after maintenance.

- Switch off the detection module via the power switch.
- Remove the left side panel. Remove the 4 screws to do this. Remove the protective grounding cable and remove the side panel.
- Remove the communication cable from the ozone generator.
- Remove the 2 hoses from the ozone generator: "O_2 in" and "O_3 out". The "O_2 in" hose connection is color-coded.
- Remove both angled FAST connectors from the ozone generator.
- Remove the screw the ozone generator is fastened to the base plate with (see arrow).
- Carefully push the old ozone generator out of the module and remove it. Insert a new ozone generator in the detection module. Mount the ozone generator in reverse order. Replace the FAST connectors with new ones when doing so.

After replacement, check the system tightness:

- Connect the detection module to the basic module.
- Switch on both modules and allow them to run in for approx. 30 min.
- Wet a strip of the indicator paper with distilled water and hold it to the ventilator on the rear of the module for approx. 30 s.
- Also check the gas outlet of the detection modules with the test strip.
- If the strip turns blue, ozone is leaking from the module. If this is the case, switch off the module, ventilate the room and check the proper connection of the hoses on the ozone generator.

✓ The detection module is ready for operation again.

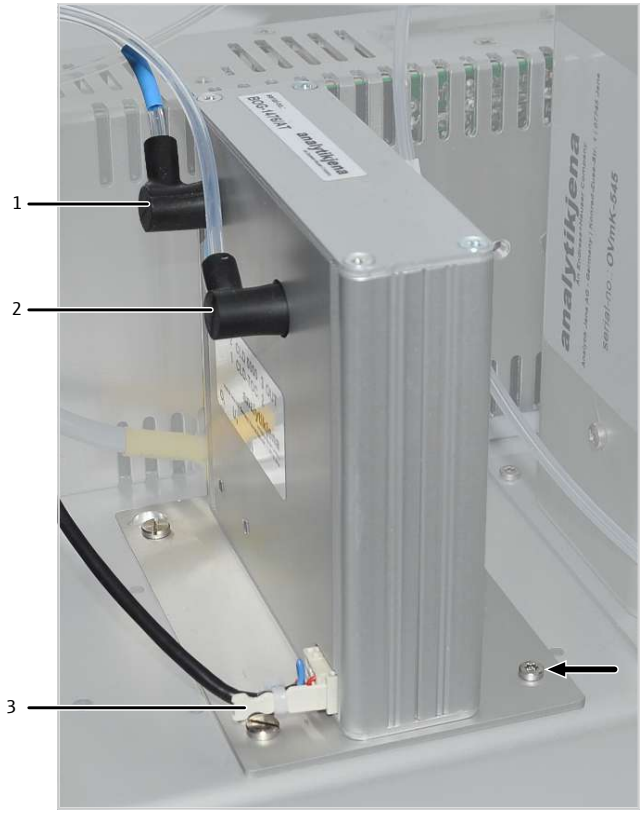


Fig. 68 Replacing the ozone generator

1 Oxygen connection (O_2 in)
2 Ozone outlet (O_3 out)
3 Communication cable (to the PCB)

Checking the function of the detection module

Check the function of the detection module after maintenance via a control measurement.

- Perform a purge measurement with a solvent, e.g., isooctane.
- Analyze a standard solution (5 mg/l TN_b) Compare the curve and area with earlier measurements.
- For resuming measuring operations: Determine the daily factor to test calibration. If the daily factor is outside of the tolerance range, the analysis system must be re-calibrated.

14.10.2 Replacing the absorber

⇨ Replace the absorber if the analysis baseline has been permanently raised. Replace the entire absorber (replacement part).

- Remove the screw connection from the absorber. Do not pull the hose out of the device!
- Pull the absorber out of the holding clamps.
- Press the new absorber into the holding clamps. Reconnect the hose.

✓ The detection module is ready for measurement again.

Fig. 69 Absorber

1 Hose 6 connection
2 Holding clamp
3 Absorber

14.10.3 Replacing the chemical ozone decomposer

WARNING

Risk of electric shock

High voltages are present in the interior of the device, which can lead to electric shock if contacted.

- Before opening: Switch off the device via the power switch.
- Disconnect the power cable from the socket.

CAUTION

Risk of burns from the thermal ozone decomposer!

- Only perform maintenance in the inside of the device when cold/allow the device to cool sufficiently.

⇨ Replace the entire chemical ozone decomposer annually. Optionally, have the decomposer replaced by the customer service department.

- Switch off the detection module via the power switch.
- Remove the left side panel. Remove the 4 screws to do this. Remove the protective grounding cable and remove the side panel.
- Disconnect the following hose connections:
  Disconnect hose 25 from the T piece.
  Unscrew hose 24 from the bottom of the ozone decomposers.
- Remove the ozone decomposer with its filter and hose 25 from the holding clamps.
  Recommendation: Remove it from the top clamp first.
- Install the new filter in the reverse order.

✓ The detection module is ready for measurement.

Fig. 70 Replacing the chemical ozone decomposer

1 Ozone decomposer
2 Hose 24
3 Filter with hose 25

14.11 Maintenance of the Cl module 5100 chlorine detector

14.11.1 Replacing the sulfuric acid and cleaning the sulfuric acid container.

WARNING

Risk of chemical burns

Concentrated sulfuric acid can cause severe chemical burns!

- Before replacing the sulfuric acid: Switch off the gas supply via the software. Risk of splashing if the gas supply is running.
- Wear protective clothing when working on the sulfuric acid container.
- Observe all instructions and specifications in the safety data sheet.

CAUTION

Risk of injury

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

- Handle glass parts with extreme caution.

The sulfuric acid absorbs the water that is produced during the combustion process. If the acid content falls below 85 % the sulfuric acid can no longer sufficiently dry the reaction gas. Chlorine values that are too low are then measured.

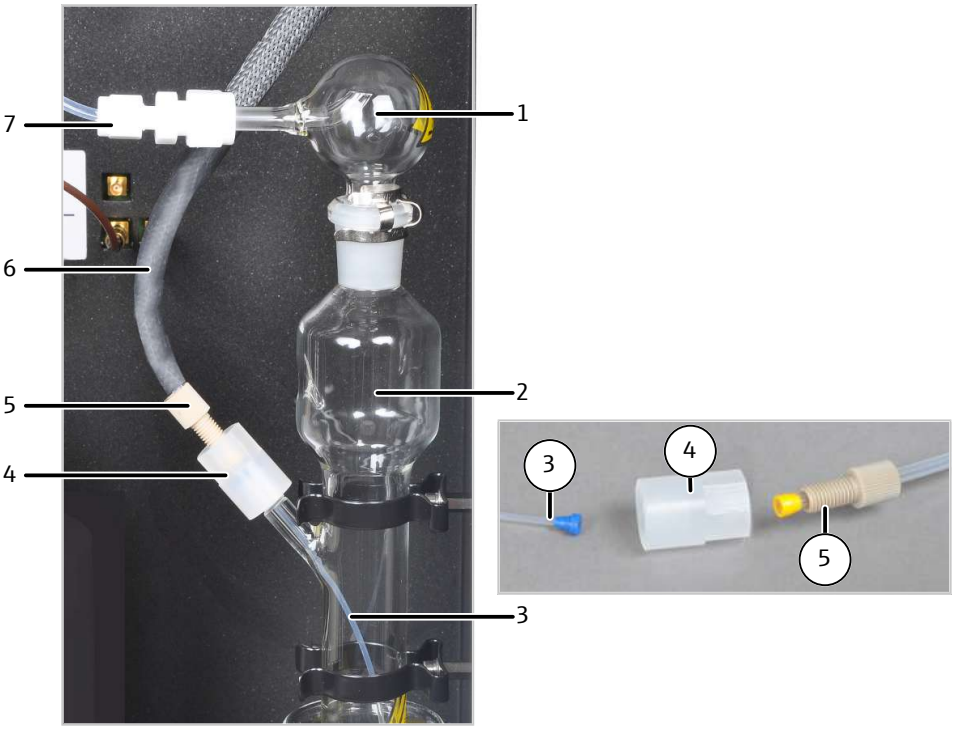


Fig. 71 Connection of the heated gas transfer lines to the sulfuric acid container

1 Safety attachment
2 Sulfuric acid container
3 Hose for measuring gas introduction
4 Connector
5 Banjo bolt
6 Heated gas transfer line
7 Measuring gas transfer to the measuring cell with PTFE connector

⇨ Replace the sulfuric acid daily. High sample throughput may require more frequent replacement.

- Exit the multiWin software and switch off the analysis system. Switch of the detection module via the switch on the rear of the device.
- Allow the heated gas transfer line to cool or wear heat-proof gloves when replacing the sulfuric acid.
  ⚠ CAUTION! Risk of burns at the ends of the heated gas transfer line! The ends can achieve temperatures of up to 100 °C during operation.
- Remove the banjo bolt from the connector, separating the heated gas transfer line from the sulfuric acid container.
- Disconnect the PTFE connection and remove hose 20 from the safety attachment.
- Carefully pull the sulfuric acid container with its remaining components out of the holding clamps toward the top and remove it from the module.
  A large beaker is suitable for safe transport and storage (e.g., 500 ml).
- For the "high sensitive" measuring cell: Remove the gas inlet tube from the detection module with PTFE screw joint and hose 20.
- Remove the safety attachment from the sulfuric acid container.
  ℹ NOTICE! The bases of the PTFE screw joints remain on the safety attachment, the hose and the gas inlet tube.

- Unscrew the connectors for the heated gas transfer line from the sulfuric acid container. Remove the thin hose from the container.
  ⚠ CAUTION! Sulfuric acid residue can still be present on the hose.
- Empty the sulfuric acid via the top opening. Dispose of the sulfuric acid.
- Rinse the sulfuric acid container and the safety attachment multiple times with ultrapure water and then rinse with ethanol or methanol:
- Rinse hose 20 including the PTFE screw joint with ultrapure water and then with ethanol or methanol.
- Dry the cleaned components, e.g., by blowing it through with an inert gas.
- Place the sulfuric acid container down somewhere safe and fill it with 20 ml of concentrated sulfuric acid.
- Install the filled sulfuric acid container in the detection module in reverse order (→ "Installation" 🖹 59).
  ℹ NOTICE! Ensure the proper fit of the conical nipples when connecting the gas transfer line and the PTFE connectors.
  ✓ The detection module is ready for operation again.

14.11.2 Maintenance of the measuring cell

WARNING

Risk of chemical burns

The electrolyte solution contains high concentrations of acetic acid.

- Wear protective clothing when replacing the electrolyte solution.
- Observe all instructions and specifications in the safety data sheet.

⇨ For the "sensitive" and "high concentration" measuring cells: Replace the electrolyte solution daily.

⇨ For the "high sensitive" measuring cell: Refill the electrolyte solution daily.
Only replace the electrolyte solution in the event of analytical problem and if crystalline deposits form.

- For replacement of the electrolyte: Empty the measuring cell. Dispose of the electrolyte solution.
- Rinse the empty measuring cell and the magnetic stirrer first with ultrapure water, then with ethanol.
- Carefully wipe the measuring cell and the magnetic stirrer with tissue to remove any possible silver chloride residue.
- Fill the measuring cell with fresh electrolyte solution:
  - "high sensitive" measuring cell: 65 ml
  - "sensitive" measuring cell: 15 to 20 ml
  - "high concentration" measuring cell: 120 ml

  ✓ The measuring cell is ready for operation again.

Also observe the following:

- If the detection module is put out of operation for multiple days, clean the measuring cell and store it in a dry place.

- Check the casing of the magnetic stirring rod for cracks regularly. If metal ions from the stirring rod come into contact with the electrolyte solution, they can disrupt the analysis.
- Due to risk of short-circuits: Prevent fluids from entering the stirring/cooling block and the plug-in contacts.

14.11.3 Maintenance and storage of the electrodes

Combined electrode

NOTICE

Risk of possible destruction of the electrode due to cleaning, abrasive or polishing agents.

The combined electrode consists of ceramic materials which is mechanically sensitive especially near the fused electrodes.

- Only rinse the combined electrode with ethanol and ultrapure water to clean it.

When handled incorrectly, the electrical connection of the combined electrode can break.

- Carefully remove the electrode from the lid of the measuring cell.
- Grip the electrode from above and pull it straight out of the lid.
- Do not pull on or jerk the side connection sleeve for the electrical connections. The connections in the sleeve will break otherwise (not visible from outside)!

Fig. 72 Correct handling of the combined electrode

Drying of electrolyte on the combined electrode can cause an irreversible reduction of its sensitivity or damage the electrode. Therefore, make sure that the electrolyte never dries on the combined electrode.

- For brief operational interruptions (one full day): Store the combined electrode in fresh electrolyte solution.
- When put out of operation for multiple days: Carefully rinse the combined electrode with ethanol and subsequently rinse it with ultrapure water. Also rinse the inner opening for the gas inlet while doing so. Wipe the combined electrode with cellulose and store it in dry condition.
- For intensive cleaning: Fill the measuring cell with ethanol. Insert the combined electrode in the measuring cell and allow the solution to stir for several hours in the detection module. Do not connect the measuring cell or the electrode to their electrical connections for this.
- Before an end point routine: Store a new combined electrode or one that has been stored in a dry place in fresh electrolyte solution for at least one hour.

- Do not touch the measuring cell and the electrode during operation (during a measurement or an end point routine). The measurement result will be falsified otherwise.
- The generator anode is located at the bottom of the measuring cell in the form of the stable silver plate (silver circle). The silver electrode wears out with increasing use. If necessary, the entire measuring cell must be replaced.

Sensor electrode

NOTICE

Risk of damage to the sensor electrode

The sensor pin and the gold contact of the sensor electrodes are sensitive to touch.

- Apply scratch protection to the sensor pin for storage.
- Rinse the sensor pin with ultrapure water before use or to clean it. Do not touch it again after this. Do not dry the pin or wipe it dry!
- Before use or to clean it, wipe the gold contact with a cloth and some ethanol. Do not touch it again after this.

Storing the sensor electrode:

- The sensor electrode can be stored in the measuring cell for several days if it is sufficiently filled with electrolyte solution.
- Clean the sensor electrode with ultrapure water before longer storage. Apply scratch protection to the sensor pin. Store the electrode in a dry place.

Reference electrode

CAUTION

Risk of injury

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

- Handle glass parts with extreme caution.

- Check the condition and filling level of the bridge electrolyte weekly. The bridge electrolyte must be clear and free of deposits or other particles.
- If necessary: Refill or replace the electrolyte solution.

Storing the reference electrode:

- The reference electrode can be stored in the measuring cell for several days if it is sufficiently filled with electrolyte solution.
- For storage of less than 1 month: Close the refill opening. Apply the empty protective cap to the electrode.
- For storage longer than 1 month: Empty the bridge electrolyte completely via the refill opening with a syringe or disposable pipette. Close the refill opening. Apply the empty protective cap to the electrode.

Putting the reference electrode back into operation after longer storage:

- Rinse the inside of the electrode with approx. 2 ml electrolyte solution.
- Fill the electrode with electrolyte solution up to the refill opening.
- Fill the measuring cell with electrolyte solution. Insert the electrode in the measuring cell and allow the solution to stir for several hours in the detection module. Do not connect the electrode to its electrical connection for this.

Platinum electrode

The platinum electrode is maintenance-free. The salt bridge of the platinum electrode has a diaphragm. The electrolyte solution must not be allowed to crystallize inside the diaphragm, otherwise it can clog. Remove the salt bridge and rinse with sufficient distilled water for longer storage intervals.

The silver electrode in the "high sensitive" measuring cell

Wipe the silver surface with cellulose after use. Otherwise, the electrode is maintenance-free. The silver electrode can show discoloration from longer use. Discoloration does not affect the service life or performance of the electrode.

14.12 Maintenance of the S module 5100 basic and S module 5100 MPO sulfur detector

14.12.1 Replacing the UV lamp

WARNING

Risk of electric shock

High voltages are present in the interior of the device, which can lead to electric shock if contacted.

- Before opening: Switch off the device via the power switch.
- Disconnect the power cable from the socket.

WARNING

Risk due to UV radiation

The UV lamp emits UV radiation that can damage eyes and skin.

- Before opening the detection module: Switch off the device via the power switch.

CAUTION

Risk of burns

The UV is hot directly after operation.

- Allow the lamp to cool before maintenance.

CAUTION

Risk of injury

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

- Handle glass parts with extreme caution.

NOTICE

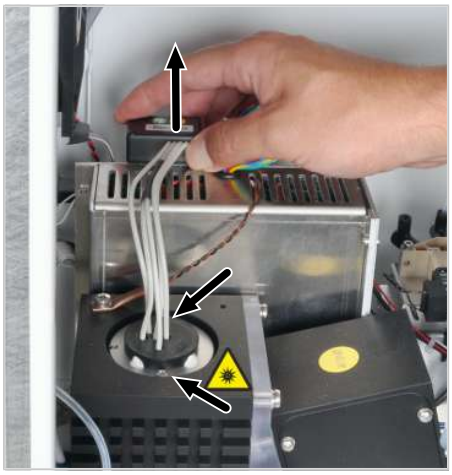
Contamination reduces the effectiveness of the UV lamp.

- Do not touch the glass of the new lamp. In particular, protect the radiation emission point made of quartz glass.
- If the glass is touched, wipe it with a lint-free cloth and pure alcohol.

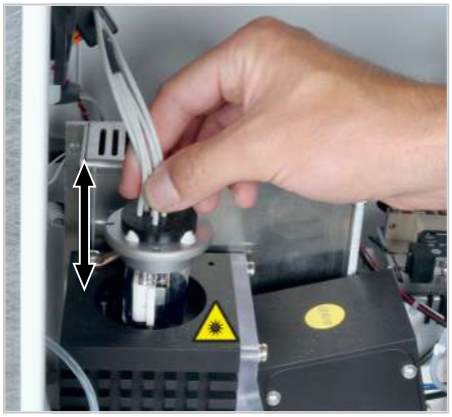
The following states indicate a defective UV lamp:

- The run-in period of the detection module takes is not complete after 30 min. The LED on the front flashes continuously.
- The measurement sensitivity is too low or the detection limit can no longer be reached.
- Check the state of the UV lamp in the **System** | **Component test** menu in the detection module tab. If a defect is indicated or the lamp is worn out, replace the lamp.

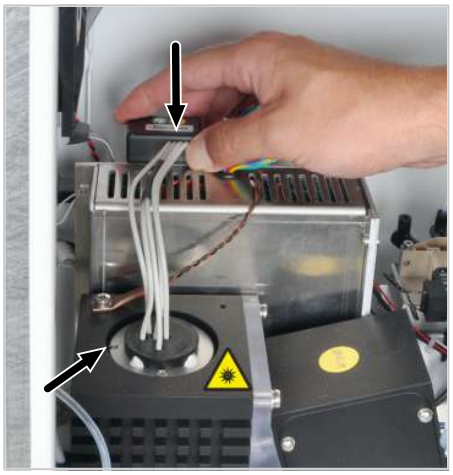
- Switch off the detection module via the power switch.
- Remove the left side panel. Remove the 4 screws to do this. Remove the protective grounding cable and remove the side panel.
  - ✓ The UV lamp is located to the left in the module (see arrow).



- Unscrew both fastening screws with a Phillips screwdriver.
- Pull the connection plug on top out of the socket.



- Carefully remove the lamp from its holder.
- Insert a new lamp in the holder.
  NOTICE! Only touch the new lamp by its base or its cable. Do not touch the glass. Do not scratch the lamp.



- Place the lamp in the proper position when inserting: The pin on the holder must fit into the groove of the lamp body.
- Fasten the new lamp with the 2 screws.
- Plug the connection plug back into the socket all the way.
- Fasten the side panel again.
  - ✓ The detection module is ready for operation again.

14.12.2 Replacing the chemical ozone decomposer

Only for the S module 5100 MPO

- ⇨ Replace the entire chemical ozone decomposer at least once annually.
- ⇨ Always replace the ozone decomposer when the smell of ozone is noticeable.
- Undo the hose from the chemical ozone decomposer. Do not pull the hose out of the device!
- Pull the ozone decomposer out of the holding clamps.
- Press the new ozone decomposer into the holding clamps. Reattach the hose.
  - ✓ The detection module is ready for measurement again.

Fig. 73 Replacing the chemical ozone decomposer

14.13 Maintenance of the coulometric sulfur detector

CAUTION

Risk of injury

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

- Handle glass parts with extreme caution.

14.13.1 Replacing the absorber

CAUTION

Skin and respiratory system irritation due to quartz wool

Quartz wool tends to form dust. Irritation can occur after breathing in or skin contact with this dust.

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

⇨ Check the absorber once per week. Replace the filling if necessary.

⇨ Replace the filling of the NOx absorber if the color has turned from light green to yellow or light brown.

⇨ Replace the filling of the HX absorber if the color has turned from metallic silver to dark gray.

▶ Remove the hose connection of the absorber tube.

▶ Remove the absorber tube from the clamps.

▶ Disconnect the FAST connector from the tube on one side. Remove the quartz wool plug.

▶ Remove the use filling from the tube.

▶ Fill fresh absorber material (silver wool for the HX absorber, ammonium iron(II) sulfate for the NOx absorber) into the tube. Reinsert the quartz wool plug. Connect the FAST connector.

▶ Carefully place the absorber tube back in the clamps.

▶ Connect the hoses to the absorber tube.

✓ The detection module is ready for operation again.

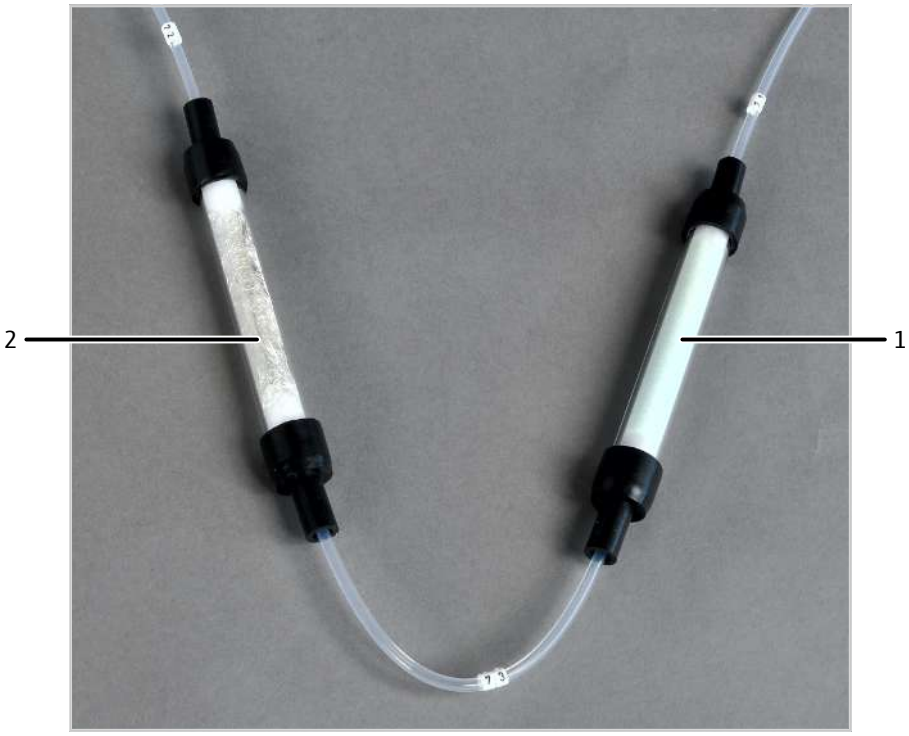


Fig. 74 NOx absorber and HX absorber

1 NOx absorber
2 HX absorber

14.13.2 Replacing the electrolyte solution

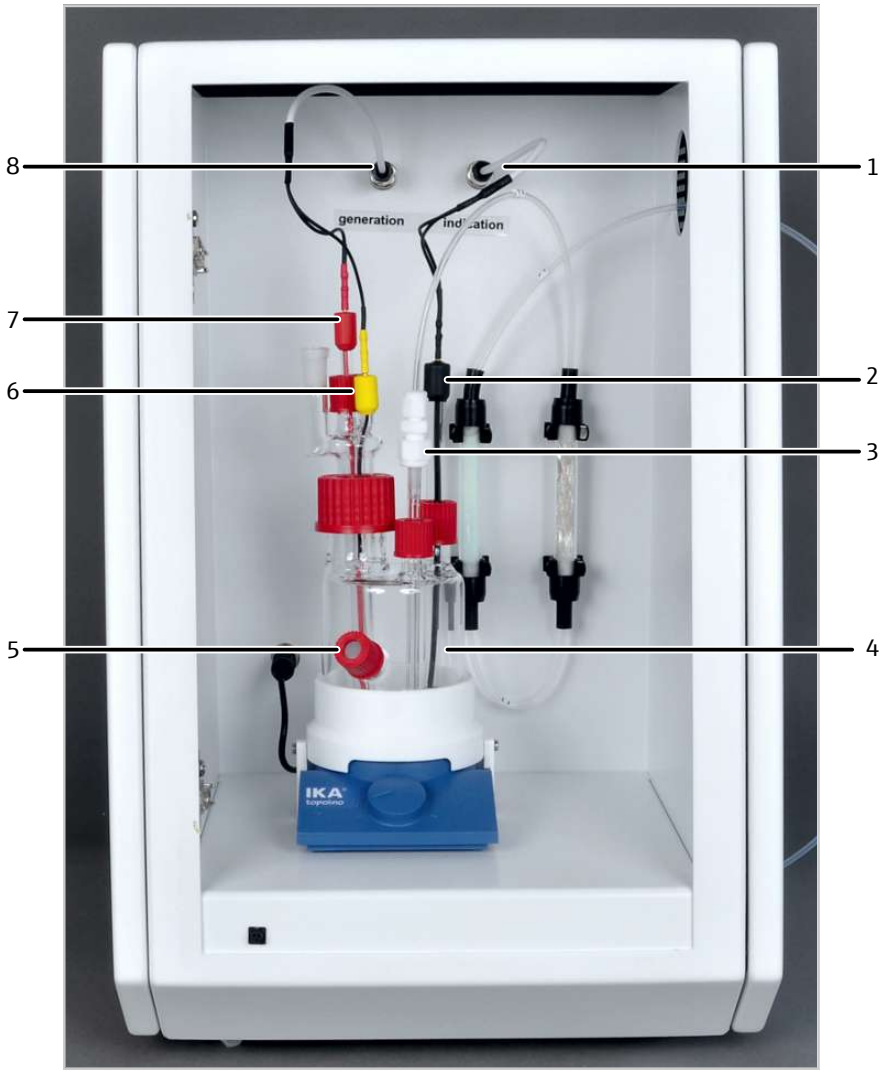


Fig. 75 Coulometric sulfur detector with measuring cell (without door)

1 Indicator electrodes connection
2 Indicator electrode (black)
3 Gas inlet
4 Measuring cell
5 Port for manual dosing
6 Anode (yellow)
7 Cathode (red)
8 Generator electrodes connection

⇨ Replace the electrolyte solution daily and when it is used up.

- Switch of the magnetic stirrer via the rotary switch.
- Switch off the detection module via the power switch.
- Disconnect the two electrode cables on the "generation" and "indication" connections.
- Disconnect hose 72 from the HX absorber.
- Remove the measuring cell from the detection module.
- Remove the electrodes and the gas inlet tube from the measuring cell. Empty the remaining electrolyte solution from the generator electrodes. Set the components down.
- Remove the electrolyte solution from the measuring cell.
- Rinse the stirring rod with ultrapure water. Rinse out the measuring cell.

- Fill the measuring cell with approx. 100 ml of fresh electrolyte solution (approx up to the manual dosing port). For creation of the electrolyte solution, see (→ "Preparing the measuring cell" 80).
- Carefully place the stirring rod back in the measuring cell.
- Reinsert the electrodes in the measuring cell. Connect the electrodes to the "Generation" and "Indication" connections.
- Place the measuring cell in the holder on the magnetic stirrer.
- Insert the gas inlet tube into the measuring cell. Connect the gas inlet tube with the HX absorber via hose 72.
- Activate the magnetic stirrer (set to stage 3 approx.).
  NOTICE! The magnetic stirring rod can damage the electrodes if the rotation frequency is set too high. Operate the rotary switch with care.
- Wait for approx. 5 min for the new electrolyte to collect in the salt bridge of the generator electrodes and for the measuring cell to establish equilibrium. Before starting any measurement, the end point routine must be performed.

  ✓ The detection module is ready for operation again.

14.14 Maintenance of the TOC detector

NOTICE

Risk of gas leaks

System tightness for the gas path from the inlet of the basic module to the outlet of the detection module is not regulated automatically.

- Always check the system tightness after performing maintenance on the detection module (→ "System tightness for TOC methods" 139).

14.14.1 Replacing the water traps

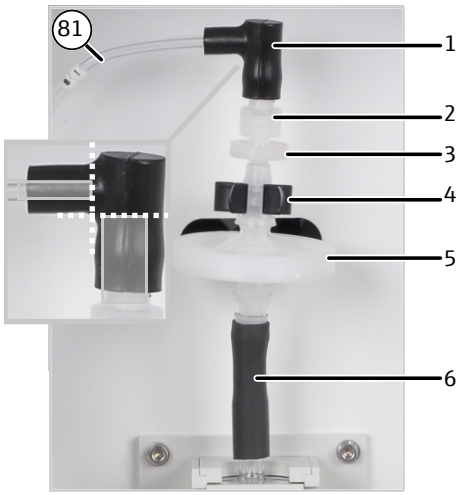


Fig. 76 Replace the water traps

1 FAST connector (angled)
2 Screw connection
3 Disposable retention filter (water trap)
4 Clamp
5 Aerosol trap (large water trap)
6 Hose connector (to the cooling block)

⇨ Replace the water traps at least every 6 months.

- Remove the hose connections from the water traps. Remove the water traps from the clamps.
- Assembly the new water traps:
  The "INLET" marking on the large water trap (aerosol trap) must be facing downward.
  The red labeling on the small water trap must face upward.
- Insert the new water traps into the clamps. The large water trap has to be positioned at the bottom.
- Connect the hose connections to the water traps.
  NOTICE! For angled FAST connectors: Do not push the connections too far into the limbs of the FAST connectors. The gas flow may be impeded otherwise.
- Check the system for leaks.

  ✓ The detection module is ready for operation again.

14.14.2 Replacing the halogen trap

CAUTION

Skin and respiratory system irritation due to quartz wool

Quartz wool tends to form dust. Irritation can occur after breathing in or skin contact with this dust.

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

NOTICE

Risk of device damage due to aggressive combustion products

When the copper wool is used up, aggressive combustion products can damage the optical and electronic components of the detection module.

- Replace the complete filling of the halogen trap as soon as half of the copper wool has turned black.

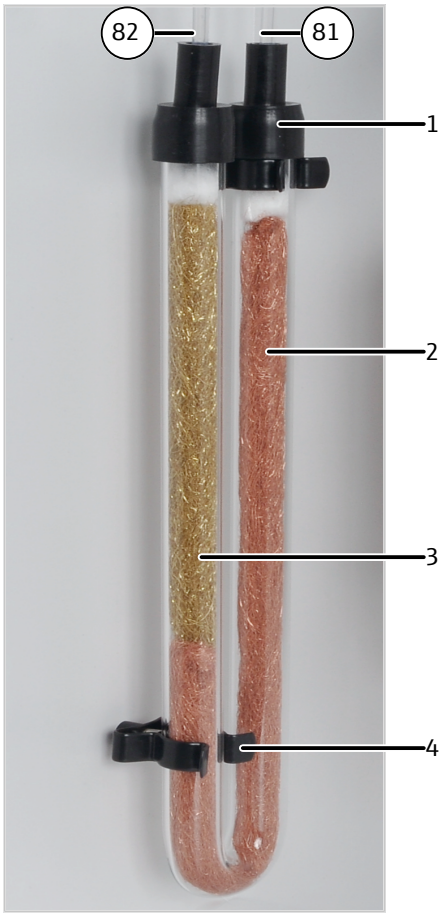


Fig. 77 Replacing the halogen trap

1 FAST connector
2 Copper wool
3 Brass wool
4 Clamp

⇨ Replace the contents of the halogen trap when half of the copper wool is discolored.

- Remove the FAST connectors from the halogen trap and remove the U-tube from the clamps.
- Remove the quartz wool plugs.
- Pull out the depleted copper wool or brass wool from the U-tube with a tweezers or a small hook.
- Inspect the U-tube for cracks.
  NOTICE! Use only completely intact U-tubes.
- If necessary, rinse the U-tube with ultrapure water and let it dry.
- Fill the U-tube with new copper and brass wool. Replace the entire contents.
  Make sure that the copper and brass wool is not compacted too much, but also ensure that there are no larger hollow spaces.
- Cover the copper and brass wool with quartz wool.
- Carefully press the filled U-tube into the clamps.
- Connect the fast connector with hose 81 to the gas inlet limb with copper wool and hose 82 to the gas outlet limb with brass wool.
- Check the system for leaks.

  ✓ The detection module is ready for operation again.

14.14.3 Regenerating the TIC reactor

WARNING

Risk of chemical burns

The TIC reactor is regenerated and cleaned with 40 % phosphoric acid. Phosphoric acid can irritate eyes, skin and mucous membranes.

- Wear protective clothing when handling the concentrated acid.
- Observe all notes and specifications in the safety data sheet.

NOTICE

Risk of leaks

A cannula that is too large will damage the septum on the septum port.

- Only use cannula with an outer diameter of 0.63 mm for the septum port.

⇨ For TIC determination or TIC determination in differential mode: The TIC reactor must be regenerated daily. How often this must be done is dependent on the TIC content of the samples. For high TIC content, regenerate the TIC reactor more than once daily.

⇨ Regeneration of the TIC reactor is also required after longer times of inactivity.

⇨ If only the TC or NPOC mode is used, regeneration of the TIC reactor is not required.

▶ Select the **System** | **Component test** menu.

▶ Select **Regeneration TIC reactor** from the list field in the **Device** tab.

▶ Click the **Regeneration TIC reactor** button.

▶ After prompt via software: Add 40 % phosphoric acid to the TIC reactor via the septum port with the supplied 5 ml syringe.

✓ The TIC reactor is drained off and purged.

14.14.4 Cleaning the TIC reactor

WARNING

Risk of chemical burns

The TIC reactor is regenerated and cleaned with 40 % phosphoric acid. Phosphoric acid can irritate eyes, skin and mucous membranes.

- Wear protective clothing when handling the concentrated acid.
- Observe all notes and specifications in the safety data sheet.

⇨ Check the TIC reactor for deposits and cracks quarterly. Cleaning is only required if TIC samples are no longer properly purged.

▶ Undo the connection between the TIC reactor and the water traps.

▶ Remove the 2 knurled screws on the cover of the cooling block. Remove the cover.

▶ Remove the FAST connector with hose 80 from the side outlet of the TIC reactor.

▶ Remove the waste hose (hose 86) to the condensate pump from the connection on the bottom of the TIC reactor.

- Remove the TIC reactor from the detection module and check it for deposits and cracks.
- Rinse the TIC reactor with ultrapure water.
- Reinstall the TIC reactor in the detection module is reverse order.
- Check the system for leaks.

  ✓ The detection module is ready for operation again.

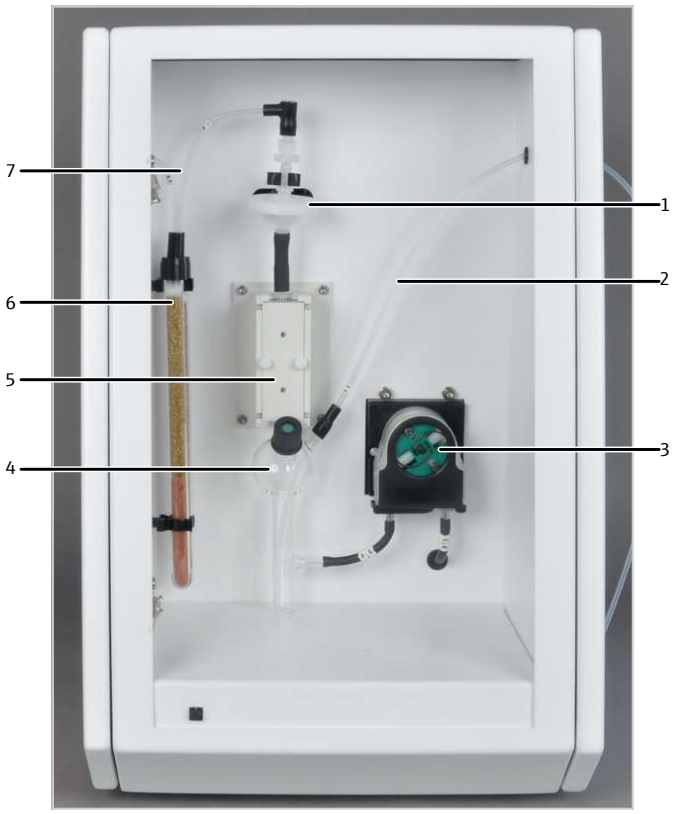


Fig. 78 TOC detector, door opened

1 Water traps
2 Measuring gas hose from the basic module (hose 80)
3 Condensate pump
4 TIC reactor
5 Cooling block (measuring gas dryer)
6 Halogen trap
7 Hose 81

14.14.5 Replacing the pump hose of the condensate pump

WARNING

Risk of chemical burns

Residue of 40 % phosphoric acid can still be found in the pump hose. Phosphoric acid can irritate eyes, skin and mucous membranes.

- Wear protective clothing when handling the concentrated acid.
- Observe all notes and specifications in the safety data sheet.

⇨ Check the condensate pump for leaks every 3 months. If any liquid is escaping from the pump hose, replace the pump hose.

⇨ When the pump body and the roller carrier are heavily damaged, they must be replaced. Inform customer service for this.

- Press the bracket on the condensate pump to the left.
- Remove hoses 85 and 86 from the pump's connections.
- Remove the guide piece with the pump hose from the pump body.
- Check the pump hose and the connections on excessive wear and cracks.
- Wipe the pump body and roller carrier with ultrapure water.
- Check the pump body and roller carrier for wear.
- Push the intact or new pump hose back into the guide piece.
  During installation, the hose clamps must be turned downwards.
- Push the hose guide into the groove on the guide piece.
- Position the guide piece with the hose around the pump body again. Press the guide piece down with one hand to do this. Move the bracket to the right until it engages with the other hand.
- Press hoses 85 and 86 back onto the metal connections of the pump hose.
- Check the system for leaks.

✓ The detection module is ready for operation again.

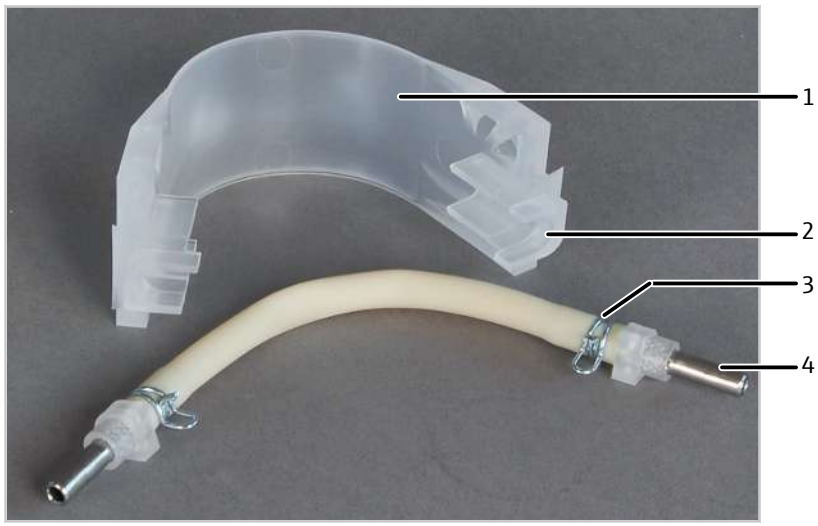


Fig. 79 Installing the pump hose in the guide piece

1 Guide piece
2 Groove
3 Hose clamp
4 Metal connection

14.14.6 Cleaning the condensation coil

CAUTION

Risk of burns from the hot furnace

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

⇨ Clean the condensation coil annually.

- Switch off the basic modules via the power switch and allow the device to cool.
- Switch off the gas supply and disconnect the power plug from the power outlet.
- Open the doors of the basic module.

- Release the fork clamp connecting the outlet of the TOC combustion tube with the condensation coil.
- Remove the fork clamp and disconnect the spherical joint connector
- Remove the FAST connector on the bottom end of the condensation coil.
- Carefully remove the condensation coil from the clamps on the combustion furnace.
- Inspect the condensation coil for deposits and cracks.
- Rinse the condensation coil with ultrapure water and dry it well.
- Reinstall the condensation coil in reverse order.
- Check the system for leaks.

✓ The analysis system is ready for operation again.

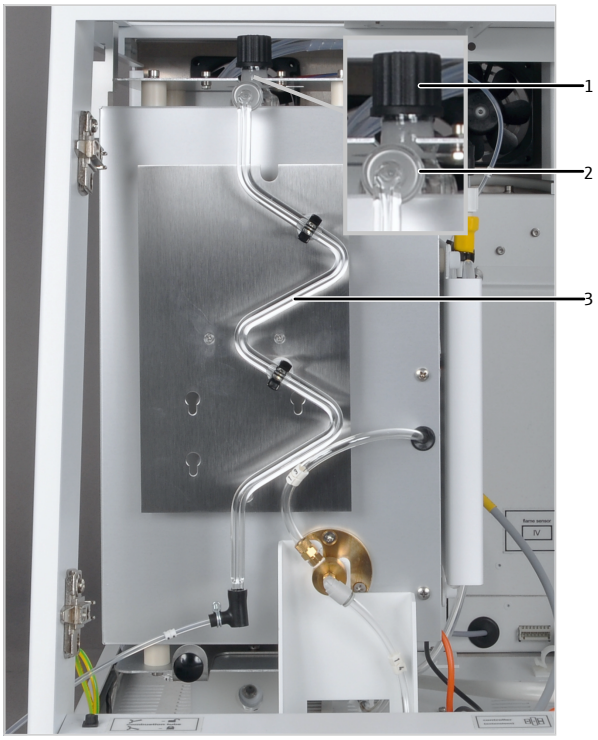


Fig. 80 Components in the basic module

1 TOC combustion tube injection port
2 Spherical joint (fasten with forked clamp)
3 Condensation coil

14.14.7 Replacing the catalyst in the TOC combustion tube

CAUTION

Risk of burns from the hot furnace

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

CAUTION

Skin and respiratory system irritation due to quartz wool

Quartz wool tends to form dust. Irritation can occur after breathing in or skin contact with this dust.

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

⇨ If the catalyst loses effectiveness, the combustion tube must be refilled. A check has to be performed after the maintenance interval has elapsed (max. 1500 injections). A software message will indicate when the maintenance interval has elapsed.

⇨ The service life of the catalyst depends largely on the samples. On average, approx. 1500 injections can be performed, more are sometimes possible. The service life can be lower with especially loaded samples, especially high salt content.

- Switch off the basic modules via the power switch and allow the device to cool.
- Switch off the gas supply and disconnect the power plug from the power outlet.
- Open the doors of the basic module and remove the top cover.
- Remove the FAST connector with hose 3 from the combustion tube.
- Unscrew the knurled head screw at the fork clamp and remove the fork clamp connecting the outlet of the combustion tube to the condensation coil.
- Disconnect the spherical joint. The condensation coil remains in the basic module.
- Remove the tube holder for holding the combustion tube.
- Carefully pull the combustion tube out of the combustion furnace toward the top.
- Remove the screw cap with septum from the combustion tube.
- Remove the used catalyst filling.
- Check the combustion tube for heavy crystallization, cracks and burst spots. Only reuse intact combustion tubes.
- Rinse the combustion tube with ultrapure water and allow it to dry.

  ✓ The TOC combustion tube is clean.

Filling the TOC combustion tube

NOTICE

Risk of devitrification of the quartz glass due to sweat

Alkaline salts in sweat from hands leads to devitrification of quartz glass when heated. Devitrification reduces the service life of the combustion tube.

- Only touch the cleaned combustion tube when wearing gloves.

- Insert approx. 500 mg of quartz glass wool in the combustion tube. Carefully press the glass wool down to a height of approx. 1 cm with a glass rod. Do not press it down too tightly.
- Carefully pour 16 g of platinum catalyst onto the quartz wool (fill height approx 4 cm).
- Cover the catalyst completely with approx 250 mg of quartz glass wool. Carefully tamp down the quartz wool.

- Fill approx. 10 g of ground quartz glass into the combustion tube (fill height approx. 2 cm).
- Cover the ground quartz glass with a piece of high temperature fiber mat (HT mat) (layer height approx 1 cm).
- Close the filled TOC combustion tube with septum and screw cap and reinstall it in the furnace in reverse order.
- Check the analysis system for leaks.

  ✓ The analysis system is ready for operation again.

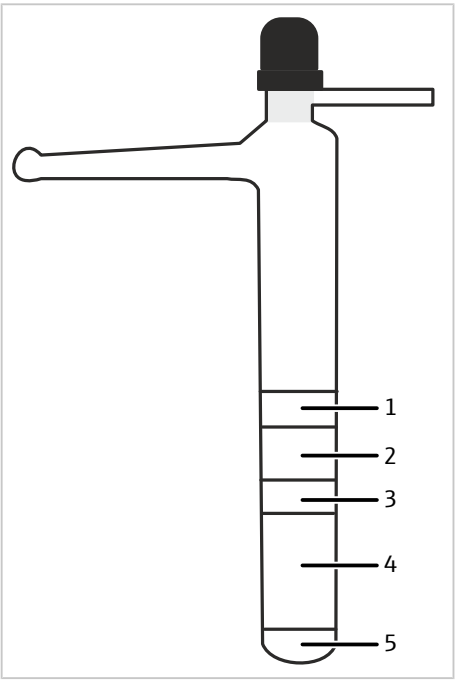


Fig. 81 TOC combustion tube

1 HT mat
2 Quartz glass fragments
3 Quartz wool
4 Catalyst
5 Quartz wool

NOTICE

The catalyst may emit gas during first heating, this can be seen as mist formation in the TIC reactor.

- Allow the catalyst to normalize during first heating for approx. 30 min until mist no longer forms. Disconnect the gas path between the TIC reactor and the water traps for this.

14.15 Cleaning the syringes

The injection syringe in the autosampler and autoinjector must be cleaned at regular intervals.

Rinsing intervals

- The syringe must be rinsed after the end of a sequence and at a minimum must be rinsed daily after the end of work.

- When analyzing samples with a complex matrix, e.g. particle-containing and inhomogeneous solutions or highly viscous liquids, rinsing after each sample is recommended in order to avoid cross contamination.

Recommended rinsing solutions

The rinsing solution should have a similar polarity to the sample and dissolve any precipitates.

Examples of samples/rinsing solutions

Sample	Rinsing solution
Petrochemicals, oils, fuels	Isooctane, toluene, xylene
Unknown samples	Absolute ethanol
General cleaning	Absolute ethanol

Autosampler

Sample	Minimum number of rinse cycles
Normal sample	3
Samples with a complex matrix	5

- Set the rinsing cycles in the method in the multiWin software.
  If required, enable automatic rinsing of the syringe in the sequence control of the configuration menu after the sample rack has been processed.

Autoinjector

Sample	Minimum number of rinse cycles
Normal sample	5
Samples with a complex matrix	10

- Remove the syringe from the autoinjector.
- Draw rinsing solution up into the syringe by hand and dispense it slowly. Repeat the process until all visible soiling has been removed.
- Insert the syringe back into the autoinjector.

Intensive cleaning

Intensive cleaning of the syringe can help with stubborn, visible soiling that cannot be removed using the above method.

- Carefully pull the plunger out of the syringe.
- Rinse the glass body and plunger with a suitable solvent or ultrapure water.
- Carefully dry the glass body and plunger. Finally, rinse both with a volatile solvent or blow out with inert gas (argon).
- When both components are clean, dry and free of particles, replace the plunger.

NOTICE! Contamination, particles and moisture can damage the Teflon seal of the plunger during assembly. This causes the syringe to leak.

Needle bocked

- Use the cleaning wire supplied with the syringe to push out the blockage.
- Then carry out intensive cleaning.

Instructions for maintaining the function of the syringe

Observe the following instructions to maintain the functionality of the syringe. Failure to do so may damage the syringe and cause it to leak.

- Do not use the syringe without liquid unnecessarily (only for aligning the autosampler or adjusting the autoinjector). Dry movement of the plunger can damage the seal.
- Do not immerse the syringe in solvents or acidic or basic aqueous solutions.
- Do not clean the syringe in an ultrasonic bath.

15 Transport and storage

15.1 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!

15.2 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.

15.3 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.

15.4 Preparing the basic module for transport and storage

Prepare the basic module for transport as follows:

- Switch off the basic modules via the power switch and allow the device to cool.
- Switch off the gas supply and disconnect the power plug from the power outlet.
- Disconnect all connection on the rear of the module.
- Remove the combustion tube (→ "Maintenance of the multi-purpose combustion tube" 🖹 124).
- Remove the auto-protection valve assembly (→ "Maintenance of the auto-protection valve assembly" 🖹 129).
- Remove the membrane dryer with holder from the combustion furnace (→ "Replacing the membrane dryer" 🖹 132).
- Remove the combustion furnace (→ "Removing and installing the combustion furnace" 🖹 139)
- Pack open hose ends in protective bags and secure them with adhesive tape.
- Close the doors of the basic module.
- Position the top cover and secure it with adhesive tape.
- Secure the maintenance flaps on the right device side with adhesive tape.
- Carefully pack the combustion furnace and additional accessories in the original packaging. Especially the glass components must be packed safely against breakage.

15.5 Preparing detection modules

CAUTION

Risk of injury

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

- Handle glass parts with extreme caution.

WARNING

Risk of chemical burns due to acid and electrolyte solution return

Concentrated sulfuric acid is used in the Cl module 5100, phosphoric acid in the TOC module 5100 and a slightly acidic electrolyte solution in the S module 5100 coulometric.

A vacuum can be created in the analysis system when the combustion furnace is cooling. This vacuum can lead to acid being drawn all the way into the auto-protection valve assembly via connection hoses and lines.

- For the Cl module 5100: Only switch off the basic module and the gas supply when the analysis system has cooled. The argon safety bypass on the auto-protection valve assembly prevents a vacuum from being created during cooling for the chlorine analysis branch. Alternatively: Remove the hose connection between the basic module and the detection module before cooling.
- For the TOC module 5100 and the S module 5100 coulometric: Before shutting down the basic module via the software, disconnection the hose connection to the detection module.

- ▶ Switch off the detection module via the power switch. Disconnect the power plug from the power socket.
- ▶ Shut off the gas supply.
- ▶ Disconnect all connections on the rear of the detection module.
- ▶ Seal the open gas connections with the ends of a short, fitting piece of hose to prevent contamination during transport.
- ▶ Remove loose components such as the absorber from the rear of the detection module and package them individually.
- ▶ If the detection module can be opened via the front door, remove all moving components and package them individually. Observe the notes for individual detection modules for this.
- ▶ Carefully package the detection module and the accessories (cables, glass components, hoses, clamps) in the original packaging.
- ▶ Add a desiccant to the package to prevent moisture damage.

15.5.1 Notes for the transport of the Cl module 5100

WARNING

Risk of chemical burns

The concentrated sulfuric acid used as a drying agent and the acetic acid electrolyte solution can cause severe chemical burns.

- Wear protective clothing when working on the sulfuric acid container and the measuring cell.
- Observe all instructions and specifications in the safety data sheets.

- ▶ Disconnect the electrodes (and the measuring cell) from the electrical connections on the inside of the rear panel of the detection module.

- Disconnect the measuring gas hose from the gas inlet tube/from the combined electrode. Ensure that the seals of the PTFE screw connection are not lost during transport.
- Disconnect the measuring cell from the exhaust hose and remove the measuring cell. Empty the measuring cell.
- Disconnect the gas transfer line from the sulfuric acid container.
- Remove the sulfuric acid container from the detection module. Empty and rinse the container (→ "Replacing the sulfuric acid and cleaning the sulfuric acid container." 145).
- Put all electrodes into their original packaging. Observe the instructions on maintenance and care of the electrodes (→ "Maintenance and storage of the electrodes" 148).
- Clean the measuring cell with distilled water and ethanol. Carefully wipe out/off the measuring cell and the magnetic stirring rod with tissue.
- Fasten the exhaust hose in the detection module, e.g., with adhesive strips.

15.5.2 Notes on transport of the S module 5100 coulometric

- Disconnect the electrode cables from the "Generation" and "Indication" connections.
- Disconnect the hose connections to the HX and NOx absorbers and remove both absorbers from the module.
- Remove the measuring cell from the module.
- Remove the electrodes and the gas inlet tube from the measuring cell.
- Empty the measuring cell, remove the magnetic stirring rod and rinse both with ultrapure water.
- Package the glass components and the electrodes in their original packaging.

15.5.3 Notes on transport of the TOC module 5100

WARNING

Risk of chemical burns

The TIC reactor can contain 40 % phosphoric acid residue. Phosphoric acid can irritate eyes, skin and mucous membranes.

- Wear protective clothing when emptying and cleaning the TIC reactor.
- Observe all notes and specifications in the safety data sheet.

- Remove the halogen trap and the water traps from the detection module.
- Remove the TIC reactor from the detection module and rinse it out.
- In the basic module: Remove the condensation coil and the TOC combustion tube after the furnace has cooled.
  ⚠ CAUTION! Risk of burns around the hot furnace!
- Package all components and connection hoses in their original packaging.

16 Disposal

When the respective service life has expired, the multi EA 5100 basic module, the detectors and sampling modules must be disposed of as electronic waste in accordance with the applicable regulations.

Waste water containing acids and sample material is generated during device operation. Dispose of the neutralized waste in accordance with the legal requirements.

Cl module 5100 electrodes

The metals used for the electrodes (platinum, silver) must not be allowed to contaminate the sewage systems, the surface or ground water or the soil. Dispose of the electrodes in accordance with applicable regulations on hazardous waste.

The chemical ozone decomposer on the absorber of the N module 5100 or S module 5100 MPO

The chemical ozone decomposer contains metal oxides. The absorber is filled with active carbon and soda lime. The used cartridges must be disposed of in accordance with local regulations.

TOC module 5100 consumables

The TOC combustion tube contains a platinum catalyst. Dispose of the used catalyst in accordance with local regulations. Analytik Jena GmbH+Co. KG will accept the special catalyst back for disposal. Please contact the customer service department (see inside front cover).

The halogen trap contains copper. Contact the responsible institution (authority or waste disposal company). There you will receive the information regarding recycling or disposal.

17 Specifications

17.1 Technical data for the multi EA 5100

General characteristics

Name on the basic module	multi EA 5100
Dimensions (W x H x D)	510 x 470 x 550 mm
Mass	25 kg

Procedural data

Digestion principle		■ Catalyst-free high-temperature incineration 2-phase process for C/N/S/Cl, EOX, EC/OC 1-phase process for AOX ■ Catalyst-assisted incineration (with the TOC module 5100) ■ Wet chemical digestion (in the TIC reactor of the TOC module 5100)
Digestion temperature		700 to 1100 °C
Measuring methods	Vertical and horizontal	TS. TN. TX. TC. EOX
	Horizontal:	AOX. EC/OC
	Vertical:	TOC. TIC. NPOC

Sampling (vertical)

TS, TN, TX, TC	Direct injection of liquids into the multi-purpose combustion tube via injection port with septum Direct injection of gases with a special, long injection needle into the multi-purpose combustion tube via injection port with septum
EOX	Direct injection of an extract into the multi-purpose combustion tube via injection port with septum
TC, NPOC	Direct injection of aqueous samples into the TOC combustion tube via injection port with septum
TIC	Direct injection of aqueous samples into the TIC reactor via injection port with septum

Sampling (horizontal)

TS, TN, TX, TC	Injection of liquids via injection port with septum (ABD) in quartz glass boats or direct transfer of solid samples in quartz glass boats into the multi-purpose combustion tube Direct injection of liquids via injection port with septum into the multi-purpose combustion tube Injection of gaseous samples with a special, flexible injection cannula via injection port with septum (ABD) into the multi-purpose combustion tube
AOX	Transfer of the loaded active carbon with a quartz container (column method, max. 18 x 6 mm columns) in the quartz glass boat into the multi-purpose combustion tube

		Transfer of the loaded, filtered active carbon without a quartz container (vibration method, with polycarbonate filters) in the quartz boat with holding-down clamp into the multi-purpose combustion tube
	EOX	Injection of an extract via injection port with septum (ABD) into quartz glass boats and transfer into the multi purpose combustion tube
	EC/OC	Transfer of the quartz-fiber filters or partial filters containing deposits in the quartz glass boat with holding-down clamp into the multi-purpose combustion tube

Sample volume	TS, TN, TX, TC, TOC	Liquids	1 to 100 µl (horizontally with ABD) 1 to 500 µl (vertically with MMS or TOC module 5100 and direct manual dosing)
		Solids	0.001 to 110 mg
		Non-pressurized gases	1 to 100 ml
		Pressurized gases	1 to 20 ml (with GSS/LPG combi module) 1 to 100 ml (with GSS module and GSS adapterbox)
		LPG	1 to 50 µl
	EOX (extract)		10 to 100 µl
	TOC (aqueous samples)		10 to 500 µl

Dosing speed (vertical)	TS, TN, TX, TC	Liquids	0.2 to 2 µl/s Recommended: 0.5 µl/s
		Non-pressurized gases	1 to 40 ml/min Recommended: 20 ml/min
		Pressurized gases	Fixed (with GSS/LPG combi module) 1 to 40 ml/min Recommended: 20 ml/min (with GSS module and GSS adapterbox)
		LPG	Solid
	EOX		0.2 to 2 µl/s Recommended: 0.5 µl/s
	TC, NPOC		100 to 700 µl/s Recommended: 350 µl/s or manually adjusted
	TIC		Manually adjusted

Dosing speed (horizontal)

Liquids, EOX	1 to 10 µl/s Recommended: 3 µl/s (with ABD + MMS or manual dosing) ABD transfer speed to furnace controlled automatically via flame sensor or software settings 0.2 to 2 µl/s Recommended: 0.5 µl/s (with Autoinjector)
Solids	Fixed/transfer speed to furnace controlled automatically via flame sensor or software settings.
Non-pressurized gases	1 to 40 ml/min Recommended: 20 ml/min
Pressurized gases	Fixed (with GSS/LPG combi module) 1 to 40 ml/min Recommended: 20 ml/min (with GSS module and GSS adapterbox)
LPG	Solid
AOX	Fixed/transfer speed to furnace controlled via software settings for the ABD

Measuring gas dryer

TS. TN. TC. EC/OC	Membrane dryer
TX. AOX. EOX	Concentrated sulfuric acid
TOC. NPOC. TIC	Condensation through Peltier cooling

Detection modules

Total nitrogen TN	N module 5100	Chemiluminescence
Total sulfur TS	S module 5100 basic and S module 5100 MPO	UV fluorescence
	S module 5100 coulometric	Coulometric titration
AOX. EOX. TX. TCl. TOX. TIX	Cl module 5100	Micro-coulometric end point titration (argentometry)
Total carbon TC. EC/OC	C module 5100	NDIR (non-dispersive infrared absorption spectrometry)
Total carbon TC. TIC. TOC. NPOC. EC/OC	TOC module 5100	NDIR (non-dispersive infrared absorption spectrometry)

Sampling modules

Liquids	Multi Matrix Sampler MMS	Auto scaling
	Autoinjector	Semi-automatic
Solids	ABD + MMS	Auto scaling
	Automatic Boat Drive ABD	Semi-automatic
Non-pressurized gases	GSS module	
Pressurized gases	GSS/LPG combi module, GSS module + GSS adapterbox	
LPG	LPG module 2.0, GSS/LPG combi module	

Process control

Control and analysis software	multiWin
Software function scope (extract)	Control of the analysis system, data logging and analysis, recalculation, data export/import, maintenance wizard, online help, real-time chart, service module, report generator, automatic leak check, self-check system

Gas supply

Oxygen	Purity		≥ 4.5
	Inlet pressure		600 kPa (6 bar)
	Consumption	Combustion	200 ml/min
		Post-combustion	200 to 400 ml/min
		Membrane dryer drying flow	Approx. 500 ml/min
Argon	Purity		≥ 4.6
	Inlet pressure		600 kPa (6 bar)
	Consumption	Combustion	100 to 200 ml/min
		Post-combustion	0 ml/min
		Pneumatic seal switch	Argon must be present
		Argon protective gas bypass (only for the Cl module 5100)	Approx. 20 ml/min

Electrical variables

Voltage	110 to 240 V +10/-5 %
Frequency	50/60 Hz
Overvoltage category	II
Fuses	T 10 AH
Number of device fuses	2
Typical average power consumption	1000 VA
PC interface	1 USB 2.0

Only use original fuses from Analytik Jena!

Environmental conditions

Temperature during operation	+21 to 35 °C
Humidity during operation	90 % at 30 °C
Air pressure	0.7 to 1.06 bar
Temperature and humidity during storage	+15 to 55 °C at 10 to 30 % humidity (use desiccant)
Maximum elevation	2000 m

Computer specifications

Graphics resolution	1280x1024 (1024x768 possible with restrictions)
CD/DVD drive	Required for software installation
Interface	1 USB 2.0
Operating system	Windows 8.1. Windows 10 (32. 64 bit)
Other	Activation of DoNetFrameWork 3.5

17.2 Technical data of the N module 5100

Procedural data

Analytical parameters	Total nitrogen TN
Detection principle	Chemiluminescence
Measuring range (N in sample)	0.01 to 10000 mg/l N
Measuring range (N absolute)	0 to 100 µg N

Electrical variables

Power supply	110 to 240 V +10/-5 %
Overvoltage category	II
Frequency	50/60 Hz
Module fuse protection	T 4.0 A H
Number of device fuses	2
Typical average power consumption	200 VA
Interface to the basic module	RS 232

Only use original fuses from Analytik Jena!

Gas supply

Oxygen 4.5	80 ml/min 400 to 600 kPa (4 to 6 bar)

General characteristics

Dimensions (W x H x D)	300 x 500 x 550 mm
Mass	13 kg

17.3 Technical data for the Cl module 5100

Methods data

Analytical parameters	AOX. EOX. TX. TCl. TOX. TIX
Detection principle	Micro-coulometric end point titration (argentometry)
Sample feed	■ In carrier gas stream (from basic module) ■ Direct injection of aqueous samples and HCl for test purposes (into the measuring cell)
Temperature control for measuring cell	Integrated cooling

Stirring mechanism of the measuring cell	Integrated magnetic stirrer (with fixed speed)
Operating range of the wide-range coulometer	3
measuring cells	■ "high sensitive" ■ "sensitive" ■ "high concentration"

"high sensitive" measuring cell

Measuring mode	Potentiometry
Measuring range (Cl absolute)	0.01 to 10 µg
Generator current	≤100 µA
Electrolyte volume	65 ml

"sensitive" measuring cell

Measuring mode	Bi-amperometry
Measuring range (Cl absolute)	1 to 100 µg
Generator current	1 mA
Electrolyte volume	15 to 20 ml

"high concentration" measuring cell

Measuring mode	Bi-amperometry
Measuring range (Cl absolute)	10 to 1000 µg
Generator current	10 mA
Electrolyte volume	120 ml

Electrical variables

Power supply	110 to 240 V +10/-5 %
Overvoltage category	II
Frequency	50/60 Hz
Module fuse protection	T 2.0 A H
Number of device fuses	2
Typical average power consumption	50 VA
Interface to the basic module	RS 232

Only use original fuses from Analytik Jena!

General characteristics

Dimensions (W x H x D)	300 x 470 x 530 mm
Mass	12 kg

17.4 Technical data for the S module 5100 (basic. MPO)

Procedural data

Analytical parameters	Total sulfur TS
Detection principle	UV fluorescence
Measuring range (S in sample)	0.01 to 10000 mg/l S
Measuring range (S absolute)	0 to 100 µg S

MPO option	TS determination in the presence of elevated nitrogen concentrations (only available with the S module 5100 MPO)

Electrical variables

Power supply	110 to 240 V +10/-5 %
Overvoltage category	II
Frequency	50/60 Hz
Module fuse protection	T 4.0 A H
Number of device fuses	2
Typical average power consumption	200 VA
Interface to the basic module	RS 232

Only use original fuses from Analytik Jena!

General characteristics

Dimensions (W x H x D)	300 x 470 x 550 mm
Mass	13 kg

17.5 Technical data for the S module 5100 coulometric

Procedural data

Analytical parameters	Total sulfur TS
Detection principle	Coulometric titration
Measuring range (S in sample)	0 to 40000 mg/l S
Measuring range (S absolute)	0 to 200 µg S

Electrical variables

Power supply	110 to 240 V +10/-5 %
Overvoltage category	II
Frequency	50/60 Hz
Module fuse protection	T 1.0 A H
Number of device fuses	2
Typical average power consumption	20 VA
Interface to the basic module	RS 232

Only use original fuses from Analytik Jena!

General characteristics

Dimensions (W x H x D)	300 x 470 x 530 mm
Mass	11 kg

17.6 Technical data for the C module 5100

Procedural data

Analytical parameters	Total carbon TC. EC/OC
Detection principle	NDIR (non-dispersive infrared absorption spectrometry)

Measuring range (C in sample)	0.1 to 10000 mg/l C
Measuring range (C absolute)	0 to 500 mg C

Electrical variables

Power supply	110 to 240 V +10/-5 %
Overvoltage category	II
Frequency	50/60 Hz
Module fuse protection	T 4.0 A H
Number of device fuses	2
Typical average power consumption	50 VA
Interface to the basic module	RS 232

Only use original fuses from Analytik Jena!

General characteristics

Dimensions (W x H x D)	300 x 470 x 530 mm
Mass	12 kg

17.7 Technical data for the TOC module 5100

Procedural data

Analytical parameters	Total carbon TC. TIC. TOC. NPOC. EC/OC
Sample feed for TIC analysis	Direct injection in the TIC reactor
Detection principle	NDIR (non-dispersive infrared absorption spectrometry)
Measuring range (C in aqueous samples)	0.2 to 10000 mg/l C
Measuring range (C in organic samples)	0.1 to 10000 mg/l C
Measuring range (C absolute)	0 to 500 mg C

Electrical variables

Power supply	110 to 240 V +10/-5 %
Overvoltage category	II
Frequency	50/60 Hz
Module fuse protection	T 4.0 A H
Number of device fuses	2
Typical average power consumption	50 VA
Interface to the basic module	RS 232

Only use original fuses from Analytik Jena!

General characteristics

Dimensions (W x H x D)	300 x 470 x 530 mm
Mass	12 kg

17.8 Standards and directives

Protection class and protection type	The device is protection class I. The housing is 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
EMC compatibility	The device has been checked for transient emissions and noise immunity. It meets the requirements for transient emissions according to ■ EN IEC 61326-1 (EN 55011 group 1, class B) The device meets the requirements for noise immunity according to ■ EN IEC 61326-1 (requirements for use in a basic environment)
Environmental and ambient influences	This device has been tested in environmental simulations under operation and transport conditions and is in accordance with the requirements in: ■ ISO 9022-2 ■ ISO 9022-3
EU directives	The device meets the requirements of the directive 2011/65/EU. The device is designed and tested in accordance with standards meeting the requirements of EU directives 2014/35/EU and 2014/30/EU. The device leaves the factory in a sound condition with regard to technical safety. To maintain this condition and to ensure safe operation, the user must strictly observe the safety and operating instructions contained in this operating manual. For accessories delivered with the device and system components from other manufacturers, the information provided in their respective operating manuals has priority.
Guidelines for China	The device contains substances subject to regulation (according to the directive GB/T 26572-2011). Analytik Jena guarantees that, if the device is used as intended, these substances will not leak within the next 25 years and therefore will not pose a threat to the environment or health within this time period.

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