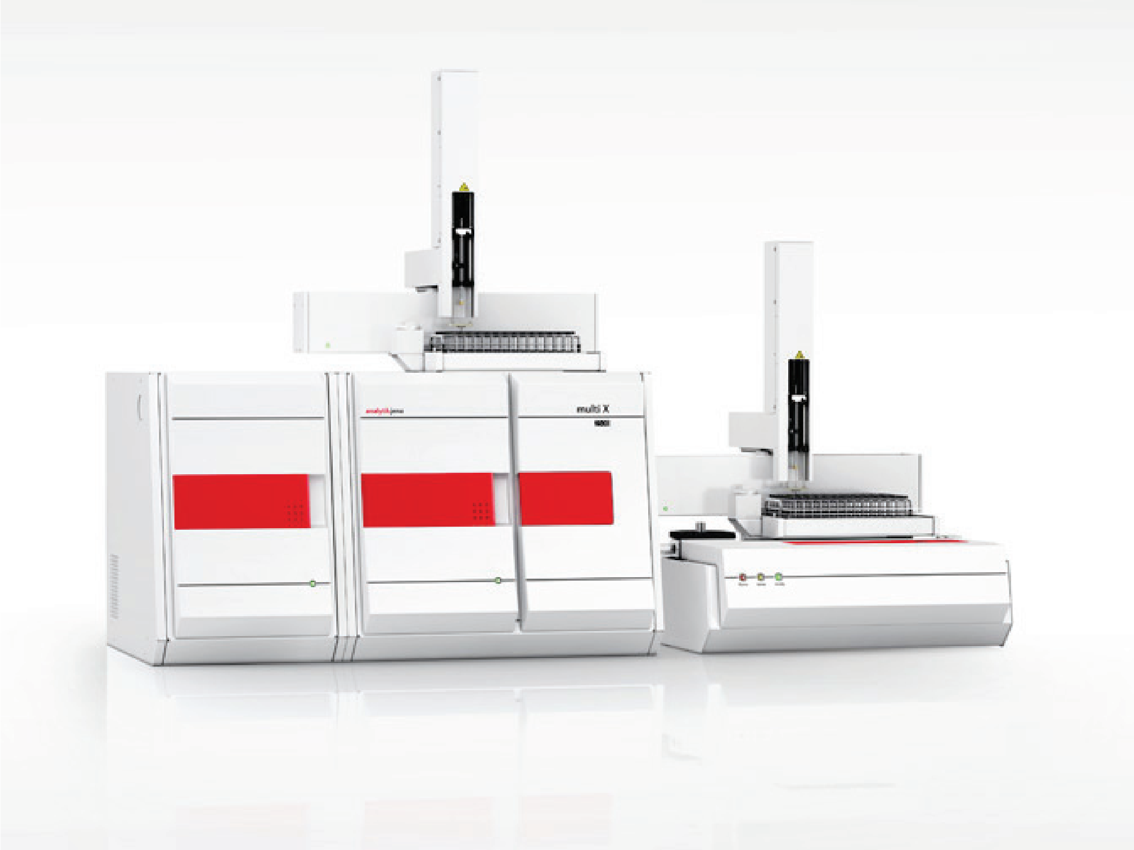
Operating Manual

multi X 2500



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

Service
Analytik Jena GmbH+Co. KG
Konrad-Zuse-Str. 1
07745 Jena · Germany
Phone + 49 3641 77 7407
Email service@analytik-jena.com

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

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1 Basic information

1.1 User manual notes

Contents

The user manual informs about the design and function of the multi X 2500 and provides the necessary know-how for the safe handling of the device and its components. Furthermore, the user manual includes information on the maintenance and servicing of the device as well as hints on potential causes for malfunctions and their correction.

Conventions

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

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

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

- Program terms are identified with small caps (e.g., the menu FILE).
- Buttons are shown by square brackets (e.g. [OK])
- Menu items are separated by arrows (e.g., FILE ▶ OPEN).

These instructions are aimed at qualified specialist users with knowledge of the analysis of sum parameters. The instructions are limited to describing the functionality of multi X 2500 and its accessories.

For the safe operation of the multi X 2500, knowledge of the operating instructions of the control and analysis software "multiWin" is also required. Basic knowledge of working with a computer is required. When using additional accessories, such as the automatic boat feeder ABD, the autosampler autoX 112 or the TOC module, the user must observe the respective operating instructions for the individual accessories.

Symbols and signal words

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

WARNING

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

CAUTION

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

NOTICE

Provides information on potential material or environmental damage.

1.2 Intended use

The multi X 2500 analyzer is used for the quantitative determination of adsorbable (AOX), extractable (EOX) or purgeable (POX) organic halogenated compounds in aqueous samples, soils and sewage sludge. The device is conceived for the analysis of drinking water, industrial water, surface water and waste water.

The analyzer may also be used to determine the total chloride content in solid and liquid organic matrices (TX/TOX). When determining the total chlorine content, the device will determine the bromine and iodine content of the sample as well. Fluorine is not detected. There is the option to upgrade the multi X 2500 by a TOC module. This allows determining the carbon content (TC, TOC, TIC) in aqueous samples.

The device must only be used with argon and oxygen as a carrier gas. For pure AOX systems and combined AOX/TOC systems, synthetic air or purified compressed air can optionally be used as a substitute for oxygen.

The multi X 2500 analyzer must only be used for the methods described in this user manual. Any other use is not as intended! Only the operator is liable for any damages that result from this.

2 Safety instructions

2.1 General notes

For your own safety and to ensure error-free operation of the multi X 2500, please read this chapter carefully before commissioning.

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

In addition to the safety instructions in this user manual and the local safety regulations that apply to the operation of the device, the general applicable regulations regarding accident prevention, occupational health and safety and environmental protection have to be observed and complied with.

References to potential dangers do not replace the work protection regulations which must be observed.

2.1.1 Safety labeling on the multi X 2500

Warnings and information symbols have been attached to the multi X 2500. These must be observed under all circumstances.

Damaged or missing warnings and information symbols can cause incorrect actions leading to personal injury or material damage! Symbol labels must not be removed or wetted with methanol! Damaged symbol labels must be replaced without delay!

Number	Warning / notice symbol	Meaning and scope of application
1		Warning against dangerous electrical voltage Warning message: Disconnect mains before opening!
2		Warning against substances hazardous to health
3		Warning against hot surface
4		Warning against corrosive substances

2.2 Requirements for the operating personnel

The multi X 2500 analyzer must only be operated by qualified specialist personnel instructed in the use of the analyzer. The instruction must also include conveying the content of this user manual and the user manuals of other system components or add-on equipment.

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

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

2.3 Safety instructions, transport and commissioning

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

Observe the following notices during transport:

- Insufficiently secured components pose a risk of injury! During transport the components of the equipment must be secured in accordance with the instructions in the user manual. The combustion furnace, auto-protection assembly, all components made of glass and the measuring cell must be removed before transporting the device.
- Only transport the analyzer in its original packaging! Ensure that the transport protections have been fitted and the analyzer is completely empty.

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

  The weight of the multi X 2500 is 25 kg. For reasons of safety 2 persons are required to transport the analyzer and must position themselves on both sides of the equipment.

  Because the analyzer does not have carrying handles, the device must be gripped firmly with both hands at the lower end. The doors must be closed.

- 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 the customer service department when registering the return. Analytik Jena must refuse acceptance of contaminated devices. The sender may be liable for damage caused by inadequate decontamination of the device.

2.4 Safety instructions - operation

2.4.1 General

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

Observe the following notes:

- The analyzer must only be operated if all protective equipment (e.g. covers, drip pans for chemicals, side walls and doors) 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.
- Modifications and extensions to the device may only be carried out after consultation with Analytik Jena. Unauthorized changes can limit the operating safety and lead to restrictions in warranty and access to customer service.
- Free access to the device switch on the rear panel of device must always be ensured during operation.
- The ventilation equipment on the multi X 2500 and the chlorine module must be in good working condition. Covered ventilation grilles or slits etc. may cause the device to break down or may cause damage to it.
- The furnace operates with temperatures of up to 1100 °C. The hot components must not be touched during or directly after the operation of the analyzer.
- Caution when handing quartz glass and glass parts. Risk of broken glass and therefore risk of injury!
- Prevent any ingress of liquids into the interior of the device. Liquids may cause a short circuit inside the housing.
- Keep all combustible materials away from the analyzer.

Protection against explosion and fire

The multi X 2500 may not be operated in an explosive environment.

- Smoking or naked flames are prohibited in the room where the multi X 2500 is operated!
- The operating personnel has to be familiar with the location of the fire-fighting equipment in the operating room of the analyzer.

2.4.2 Safety instructions - electrical equipment

Contact with live components may cause death, serious injury or painful electrical shock. Life-threatening electrical voltages may occur in the right part of the multi X 2500 behind the control electronics cover.

Observe the following notes:

- Only connect the analyzer and the chlorine module to properly earthed power outlets conforming to the voltage rating on the type plate. The protective effect must not be invalidated by the use of an extension line which does not have a protective conductor.
- Analyzer, chlorine module and any sample feeding module must only be connected to the mains when they are electrically switched off!
- Electric connection cables between the analyzer and the chlorine module or sample feeding modules must always be switched off before being connected or disconnected. Only connect the chlorine module using the designated interfaces of the multi X 2500.
- Before opening the analyzer it must be switched off using the device switch and the mains plug must be removed from the power outlet! Work on the electronics (behind the cover) may only be carried out by the customer service of Analytik Jena and specially authorized technicians.
- The electrical components must be checked regularly by a qualified electrician. Any defects, such as loose connections, faulty or damaged cables, must be repaired without delay.
- The analyzer must be switched off immediately using the device switch (on the rear of the housing) and the power supply disconnected from the mains if there is any interference with the electric components.

2.4.3 Safety instructions for compressed gas containers and systems

The carrier gas is taken from compressed gas cylinders or local compressed gas systems. Make sure that the carrier gas has the specified purity (refer to "Technical data" on p. 123)! Work on compressed gas cylinders and systems must only be carried out by individuals with specialist knowledge and experience in compressed gas systems. Unauthorized assembly and installation are not permitted!

Observe the following notes:

- The safety instructions and guidelines for operating compressed gas cylinders or compressed gas systems that apply at the operating location must be strictly complied with.
- Compressed air hoses and pressure reducers may only be used for the assigned gases.
- Pipes, hoses, screw connections and pressure reducers for oxygen must be kept free from grease.
- All pipes, hoses and screw connections must be checked regularly for leaks and externally visible damage. Leaks and damaged must be repaired without delay.
- The gas supply must be closed prior to inspections, service and repairs! After successful repair and maintenance of the components of the compressed air cylinders or system the analyzer must be checked for sound operation prior to recommissioning!

2.4.4 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 the instructions in the EC safety data sheets provided by the manufacturer must be complied with.

- Special care must be taken when handling concentrated acids.

  Concentrated sulfuric acid is used as a desiccant in the multi X 2500.

  The electrolyte solution is prepared using concentrated nitric acid and acetic acid (glacial acetic acid). The preparation also includes the use of the two organic substances methanol and thymol which are hazardous to health.
- Caution! Acetic acid vapors may occur during chlorine analysis. These can strongly irritate the human respiratory tracts.
- When working with quartz wool avoid the creation of dust! Inhaled dust might cause irritation to respiratory tracts.

2.4.5 Safety instructions: service and repair

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

Independent servicing can lead to maladjustment or damage to the analyzer. Therefore, the operator may generally only carry out the tasks listed in the chapter "Maintenance and care".

Observe the following notes:

- The exterior of the analyzer may only be cleaned with a damp, not dripping, cloth after the analyzer has been switched off.

- Any service and repair work at the analyzer may usually only be carried out in the switched-off condition (unless stated otherwise).
- Service tasks and the replacement of system components must only be carried out after the system has sufficiently cooled down.
- The gas supply must be closed prior to inspections, service and repairs!
- Use only original spare parts or consumables. Only these parts are tested and guarantee safe operation. Glass parts are wearing parts. Damage to these wearing parts is not included in the warranty. The notes in the chapter "Maintenance and care" must be observed.
- All protective equipment must be reinstalled correctly immediately after completion of the service and repair work and be checked for operation!

2.5 Safety equipment / Behavior during emergencies

If possible, cut the gas supply immediately in hazardous situations or in case of accidents. If there is no immediate risk of injury, put the equipment switches of the multi X 2500 and the chlorine module to position "0" and/or unplug the mains plugs from the power outlets.

When working with the optional version:

- There is a risk of injury from sulfuric acid! In case of an emergency shutdown, the vacuum which builds up during the cooling down period may draw sulfuric acid into the gas transfer pipes and the auto-protection assembly. If possible, disconnect the gas transfer line from the sulfuric acid container in the chlorine module before cutting the gas supply.
- After an emergency shutdown, always observe the instructions in the section "Restarting the analyzer after an emergency shutdown" on page 50 prior to recommissioning the analyzer!

3 Function and layout of the multi X 2500

3.1 Principle of operation

The multi X 2500 allows determining organically bound halogens in a sum parameter analysis using the AOX, EOX or POX method. These methods generally comprise three main steps which basically only differ in the first step which is the sample preparation.

1. Enrichment of organic halogen compounds and separation of inorganic halogenides:
   - by adsorption on activated carbon (AOX),
   - by extraction with organic solvents (EOX), or
   - by stripping in the carrier gas stream (POX).
2. Mineralization of the organic compounds:

   The loaded activated carbon or an aliquot of the extract or the loaded carrier gas is introduced to the reactor, pyrolized in the argon stream if applicable and combusted in an oxygen stream at a temperature of at least 950 °C into hydrogen halide, carbon dioxide and water:

   $R\text{-}X + O_2 \rightarrow HX + CO_2 + H_2O$ (1)

   R = residual hydrocarbon

   X = Cl, Br, I

   Oxygen and argon are both used as carrier gases at the same time. The pyrolysis gases are dried with concentrated sulfuric acid.
3. Detection of the hydrogen halides (HCl, HBr, HI) via micro-coulometric titration (HF is not determined).

   This process will take place in an acetic acid medium in accordance with the following reaction equation (2):

   $Ag^+ + X^- \rightarrow AgX$ (2)

The silver ions needed for halide precipitation are produced electrolytically on a silver anode. When the halide which was produced during combustion has been decomposed the concentration of silver ions will rise in the electrolyte. This point in the process is called the titration end point. The titration end point is determined using a pair of polarized indicator electrodes (biamperometrically) or a potentiometric indicator electrode. Based on the Faraday law, the amount of charge used until the analyte is completely decomposed is used to calculate the amount of halides. The result of this calculation is indicated in relation to chloride.

TX determination

The sample preparation described under item 1 is omitted when determining the total chlorine content (TX). The samples are introduced to the analyzer directly and combusted there. The halide ion content is determined in relation to chloride.

TOC determination

There is the option to use the multi X 2500 for determining the TOC in aqueous samples. For this purpose, the analyzer must be upgraded with the TOC module. System setup and execution of the TOC measurement are described in the separate user manual of the TOC module.

3.2 Measuring methods

3.2.1 General information

The methods for AOX, EOX and POX determination are specified in various different international standards and regulations (e.g. ISO 9562). All instructions and definitions contained in these documents with regard to sampling, sample preparation and execution of the analysis must be observed.

3.2.2 AOX determination

The parameter AOX describes the sum of adsorbable organically bound halogens (chlorine, bromine, iodine). The halogens are adsorbed onto activated carbon under defined conditions ($pH < 2$ with HNO_3) from a water sample or a solid sample (sludge and sediments). The loaded activated carbon is then combusted in the oxygen stream. In the chlorine module, the hydrogen halides which are produced are absorbed in the electrolyte solution. The halide ions are determined argentometrically.

Two possible methods for absorbing the activated carbon are:

- Column method
- Batch method

Another possible method is the stirring method (or carbodisc method) which is not described in further detail in this manual.

Column method

The prepared water sample is passed through columns packed with activated carbon. During this process, the organically bound halogens are adsorbed onto the activated carbon. Inorganic halides are washed out using a nitrate solution.

The samples for the column method can be prepared with the automatic sample preparation modules APU 28 connect S, APU 28 connect SPE and their flexi versions, APU*sim* or AFU 3 (with the column method kit).

Batch method

Activated carbon and nitrate solution are added to the prepared sample in an Erlenmeyer flask. The mixture is then shaken for one hour. The suspension is then passed through a filtering system. The filter cake is washed with nitrate solution.

The sample for the batch method can be prepared using the automatic filtration unit AFU 3.

3.2.3 EOX determination

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. Hexane, heptane, pentane or petroleum ether are used for extraction.

After extraction, the extract is combusted in the argon/oxygen stream. The hydrogen halides produced will then be absorbed in the electrolyte solution before the halide ions are determined argentometrically.

3.2.4 POX determination

The parameter POX is the sum of predominantly volatile organically bound halogens (chlorine, bromine, iodine) which are purged from an aqueous sample under defined conditions. These are thus separated from the aqueous phase without further enriching in the gaseous phase. The gas used for purging is argon.

After purging, the separated material is combusted in the argon/oxygen stream. The hydrogen halides produced will then be absorbed before the halide ions are determined argentometrically.

3.3 System design

Standard version

The standard version of the analyzer is composed of the following components:

- multi X 2500 with combustion system
- chlorine module (detector)
- PC with multiWin software for device control and data analysis

The standard version of the multi X 2500 uses the vertical operating mode and the "sensitive" measuring cell for determining the sum of adsorbable organic halogenated compounds (AOX). There is the option to equip the multi X 2500 with the autosamplers autoX 36, autoX 36d or autoX 112.

Optional versions

The optional horizontal operating mode requires an extension kit with multi-purpose combustion tube and an additional boat feeder.

Self-check system

A self-check system (SCS) is integrated into the multi X 2500. The SCS is a combination of hardware components and software functions that independently ensure the error-free operation of the entire analysis system. The SCS verifies all parameters relevant for the device's safety and the quality of the analysis several times per second (e.g. gas flows, temperatures, pressures, signal drifts, cooling time, flame value etc.).

3.4 Layout of the multi X 2500

The basic device of the multi X 2500 comprises the following components:

- Electronics/internal device control
- Gas supply with gas box
- Combustion system
- Measurement gas drying (standard version)
- Measuring gas transfer

All components of the basic device which are operated or serviced by the user can be reached via the front doors. It is possible to unhinge and reinsert the front doors to allow for better access to the device interior. The mains connection, the gas terminals and the interface for the system components are located on the rear of the device.

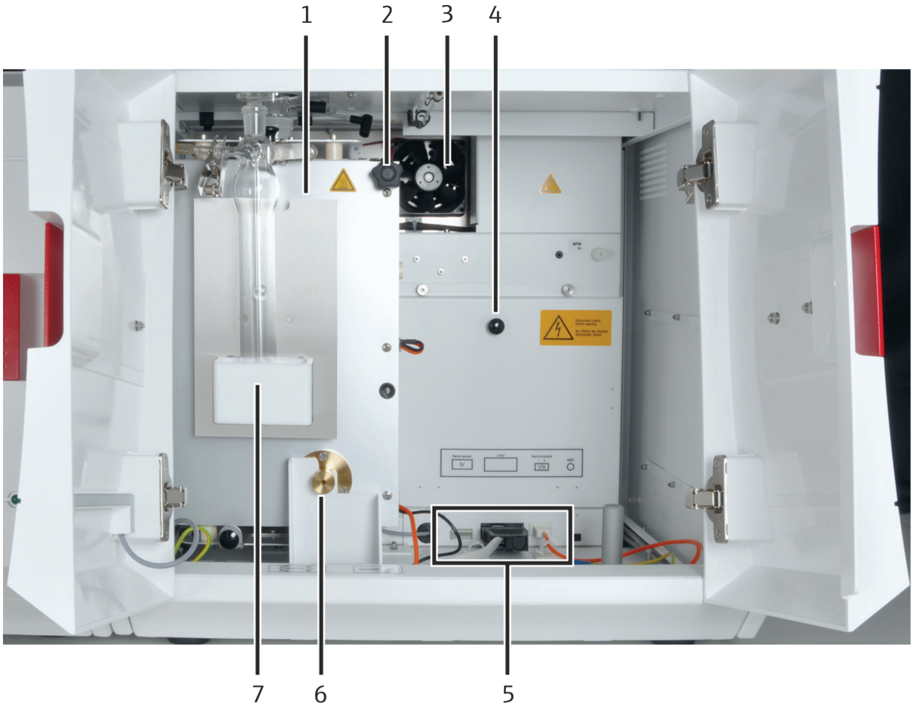


Fig. 1 **Front view (with open doors)**

1	Furnace	5	Slots
2	Knob for tilting the furnace	6	Tilt device furnace
3	Fan	7	Collection tray with sulfuric acid container (vertical) and plug
4	Control electronics cover		

3.4.1 Electronic components

LED display

A green LED is fitted to the left-hand door of the multi X 2500. After loading the multiWin software, the LED illuminates to indicate the operational readiness of the basic device.

Fig. 2 Front view with status LED

Mains connection and interfaces

The mains connection with mains switch and the interfaces for connecting the system components are located on the rear of the device.

The computer is connected via a USB port. The interfaces for connecting the autosampler and the detector module are RS 232 ports.

Fig. 3 Rear view multi X 2500

1	Interfaces (see Fig. 4)	3	Mains connection, equipment switch
2	PC connection via USB		

The equipment fuse and the mains connection are located below the device's mains switch. The rating plate is also attached there.

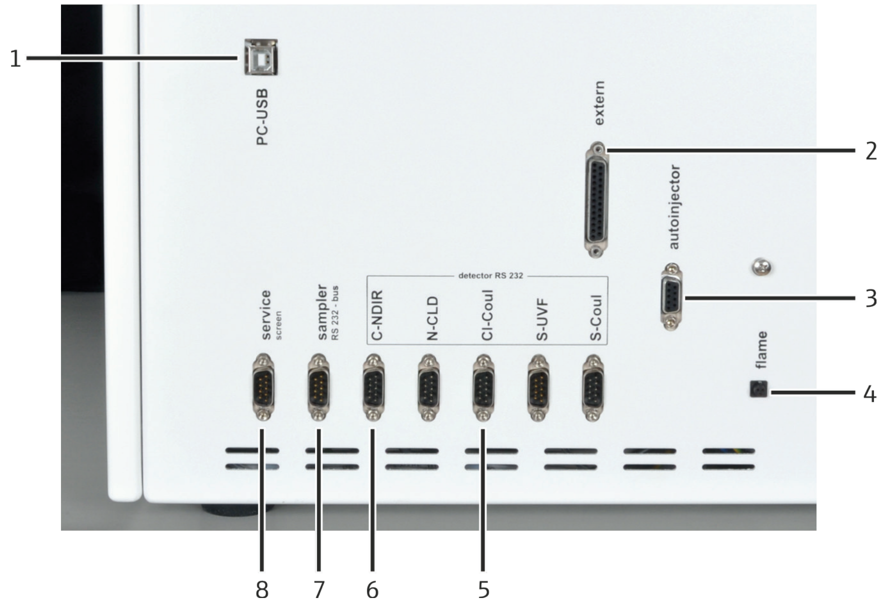


Fig. 4 **Electrical interfaces on the rear of the multi X 2500**

1 USB connection to the control PC
2 External connection (to the pre-combustion adapter)
3 Auto-injector connection
4 Flame sensor connection (to the ABD)
5 Connection Cl module
6 Connection TOC module
7 Connection sampler (RS 232 bus)
8 Connection service

Type plate

The type plate contains the following information:

- company name and complete address of the manufacturer
- type designation of the device
- serial number
- electrical connection data
- Conformity markings

Device control, interfaces in the device

The control electronics are on the right-hand side of the rear panel of the basic device behind the cover. The internal device control is used for power supply, controlling the individual components of the basic device and the communication with the computer and other connected system modules.

The electrical connections of the combustion furnace, the flame sensor and the thermocouple are located right under the control electronics' cover. The connections are only accessible in the vertical installation position of the combustion furnace.

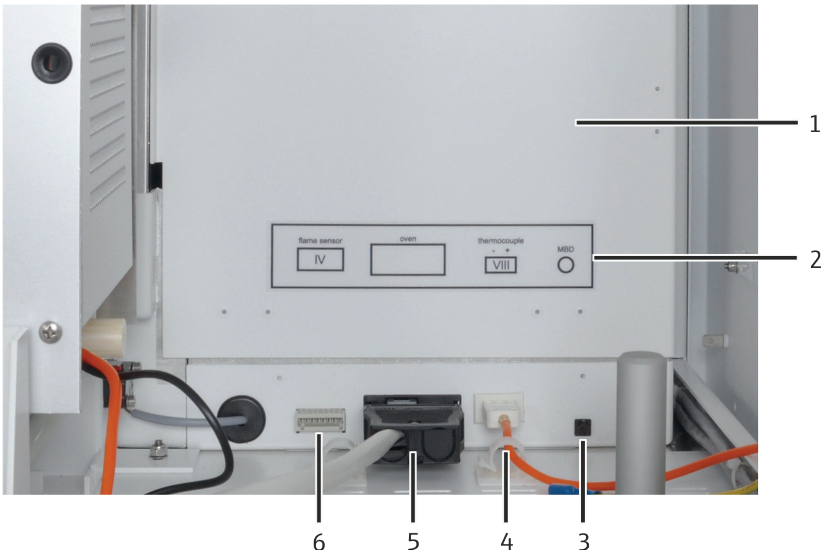


Fig. 5 **Device control and interfaces in the device interior**

1	Control electronics (behind the cover)	5	Interface combustion furnace
2	Marking interfaces	6	Interface flame sensor (optional, in horizontal operation with ABD)
3	Interface manual boat feeder MBD		
4	Interface thermocouple		

Connections
Auto-protection assembly

When using the multi-purpose combustion tube the measuring gas is routed through the auto-protection assembly. The connections for the auto-protection assembly and the heated gas transfer line are located in the frame behind the doors. The toggle switch for opening and closing the pneumatic seal in the auto-protection assembly is also located there.

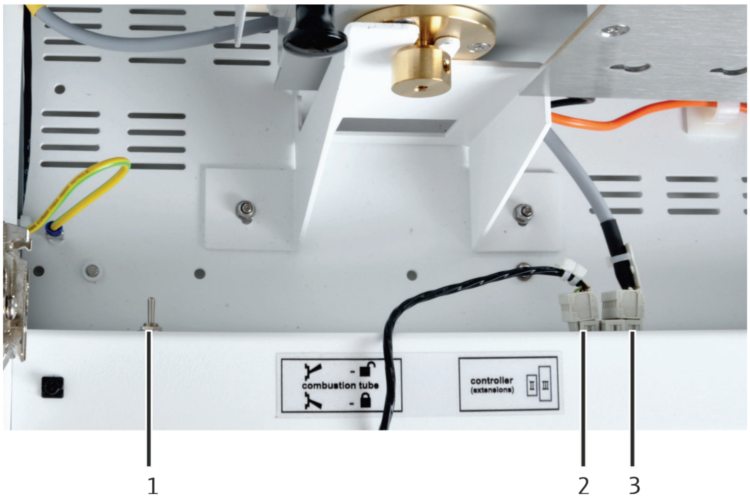


Fig. 6 **Connection for the auto-protection assembly (top view)**

1	Toggle switch for opening and closing the pneumatic seal in the auto-protection assembly
2	Heating connection for gas transfer line
3	Connection auto-protection assembly

3.4.2 Gas supply / measuring gas transfer

Gas connections at the rear of the device

The gas connections are located on the rear of the device. The carrier gas supply must be connected via the connection hoses included in the scope of delivery (6 x 4 mm) to the connections "IN O_2" or "IN Ar".

When connecting the automatic or manual boat feeder (only in horizontal operating mode) the gas supply for these two sampling systems is provided via the "OUT ABD" connection of the multi X 2500.

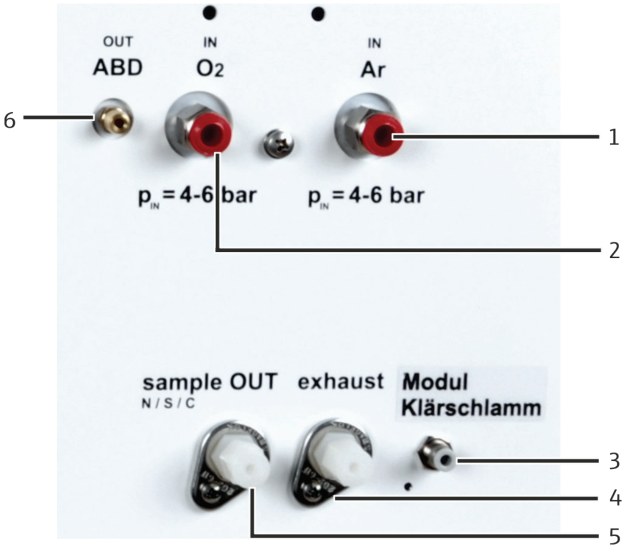


Fig. 7 **Gas connections at the rear of the device**

1	Gas inlet for Argon (Ar)	4	Purge gas outlet of the membrane dryer
2	Gas inlet for oxygen (O_2)	5	Gas outlet (not used)
3	Gas inlet pre-combustion adapter	6	Gas outlet Ar/O_2 to the ABD/MBD

When using the TOC module, the "OUT ABD" gas outlet (6 in Fig. 7) is also used for the NPOC purge gas flow.

Gas box

In the multi X 2500, the two process gases oxygen and argon are controlled via the gas box. The gas box is located on the left side inside the device.

Fig. 8 **Gas box**

Open combustion tube (standard version)

The O_2 supply on the open combustion tube is effected via the oxygen sluice inserted into the opening of the combustion tube. The gas outlet of the combustion tube is directly connected to the sulfuric acid container (vertically) using a forked clamp.

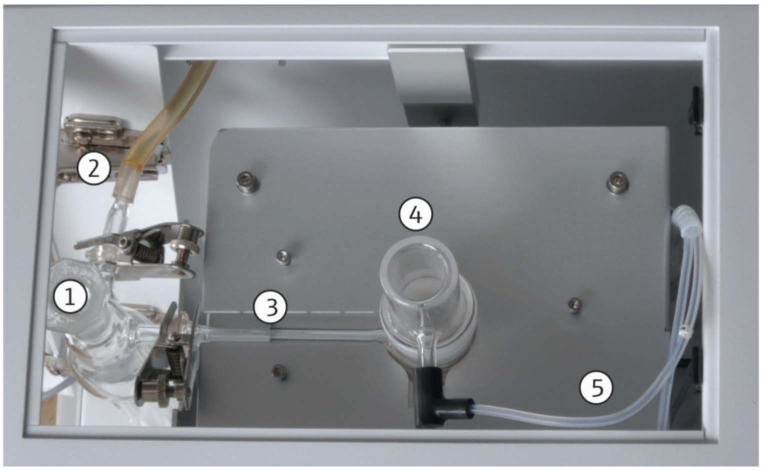


Fig. 9 **Gas connections on the open combustion tube (vertical operating mode, top view)**

1	Sulfuric acid container (vertical) with plug	4	Oxygen sluice
2	Spherical joint adapter with hose for connecting the chlorine module	5	O_2 connection – hose 3 (open combustion tube)
3	Gas outlet (open combustion tube)		

Multi-purpose combustion tube (optional)

The multi-purpose combustion tube requires oxygen as the combustion gas and argon as the carrier gas. The composition of the gas mixture for optimum gas digestion is controlled via the flow management system (FMS).

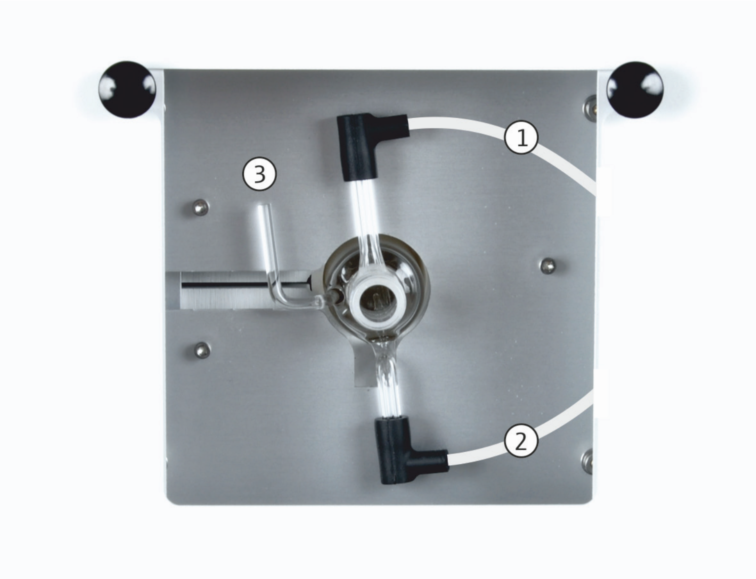


Fig. 10 **Connections on the multi-purpose combustion tube in horizontal mode (top view)**

1	Ar connection – hose 4	3	Flame sensor connection (optional)
2	O_2 connection – hose 3		

3.4.3 Combustion system

The combustion furnace is a resistor-heated furnace for combustion temperatures between 700 °C and 1100 °C. Its tilt device is easy to operate and allows the user to quickly change between the operating modes.

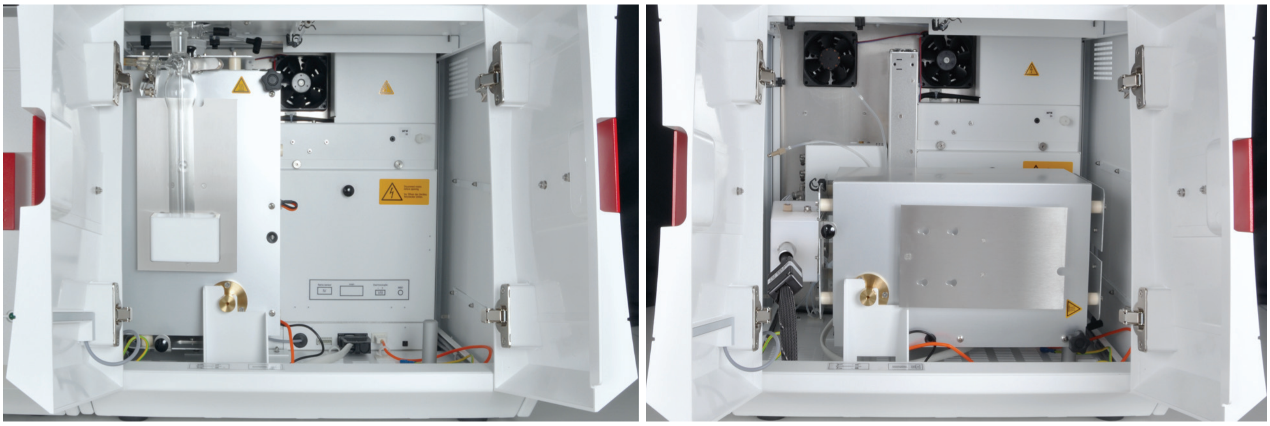


Fig. 11 Combustion furnace in the vertical and horizontal operating mode

Open combustion tube

The default setup of the multi X 2500 basic device uses an open combustion tube. This combustion tube is used for the following analyses:

- AOX determination in vertical operating mode (recommended)
- POX determination

A quartz glass wool plug is located in the bottom of the combustion tube. The quartz glass wool filters ashes and soot from the measuring gas stream. When inserting the quartz container, the plug cushions and prevents damaging the quartz glass.

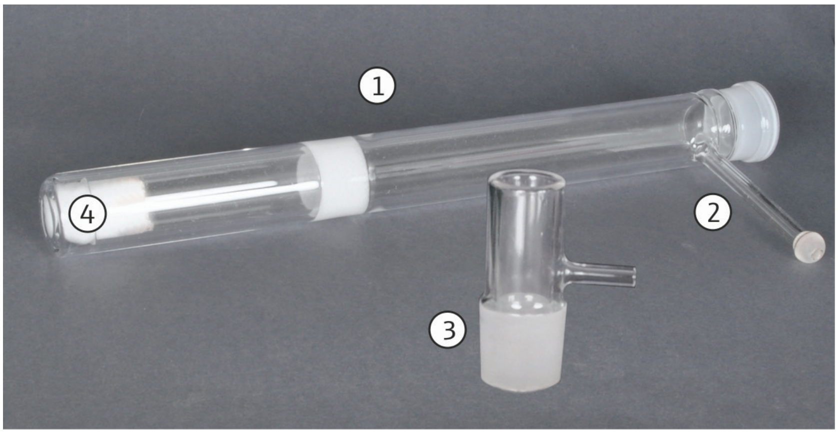


Fig. 12 Open combustion tube

1	Combustion tube	3	Oxygen sluice
2	Gas outlet with spherical joint	4	Quartz wool plug

The combustion tube can take a maximum of 40 quartz containers (depending on their size and the method employed). When reaching the maximum amount, the user must wait until the combustion tube has cooled down and empty it.

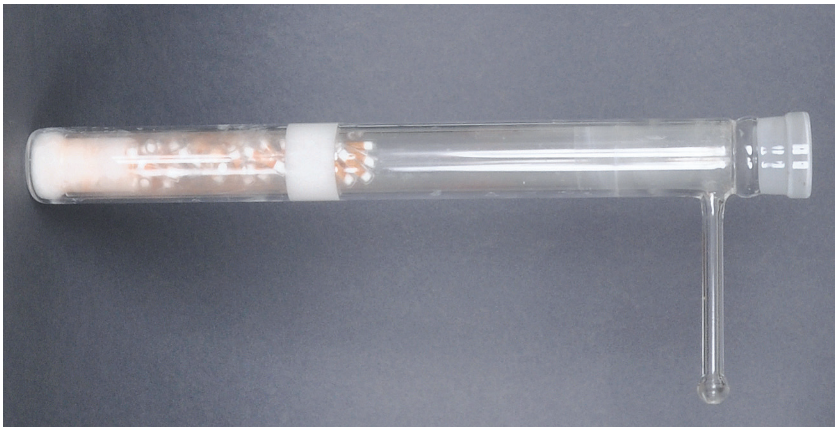


Fig. 13 Combustion tube filled with used quartz containers

Multi-purpose combustion tube

The multi-purpose combustion tube is used for the following analyses:

- AOX determination in horizontal operating mode
- EOX determination
- TX determination

The gas connections are located on the collar of the combustion tube.

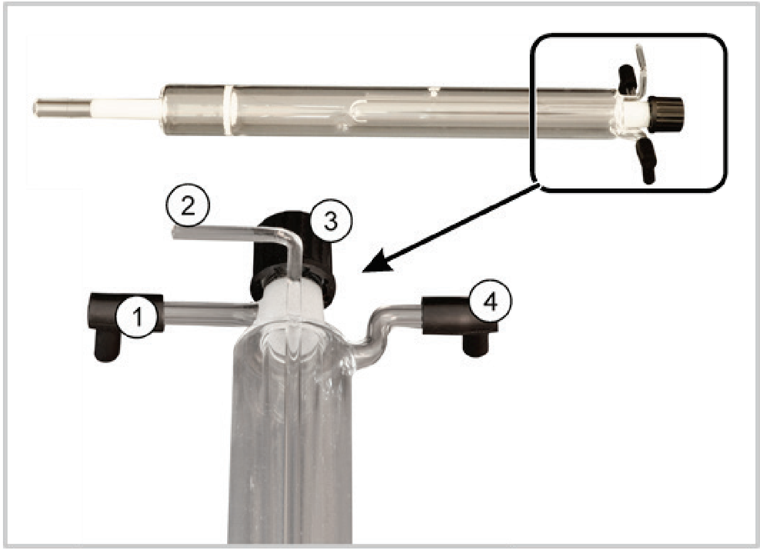


Fig. 14 Multi-purpose combustion tube

1	Argon supply connection	3	Screw cap with septum (for EOX determination only)
2	Connection for flame sensor	4	Oxygen supply connection

The connection between the combustion tube and the additional measuring gas drying or downstream measuring gas path is implemented via the auto-protection assembly.

The flame sensor is a recommended option for operating the automatic boat feeder (ABD) in horizontal mode. The flame sensor controls the speed of the boat feed to avoid an elevated soot formation inside the combustion tube. It can be used for samples with a high percentage of organic compounds (EOX and TX determinations).

3.4.4 Measuring gas drying in the standard version

Concentrated sulfuric acid is used for drying the measuring gas. The sulfuric acid container (vertical) has a capacity of 40 ml. This capacity is enough for processing up to 100 samples. In the standard version, the sulfuric acid container is attached to the furnace and directly connected to the open combustion tube via a spherical joint.

The sulfuric acid container (vertical) and the measuring cell in the chlorine module are connected via a spherical joint adapter with a hose. The intake pump in the chlorine module transfers the measuring gas into the measuring cell.

Fig. 15 Sulfuric acid container (vertical) in the standard version

1	Gas outlet combustion tube	3	Measuring gas outlet to the chlorine module
2	Sulfuric acid container (vertical) with plug		

There is a particular sulfuric acid container that is mounted to the chlorine module which can be used with the optional multi-purpose combustion tube.

3.4.5 Auto-protection assembly (optional)

The multi-purpose combustion tube requires the use of the auto-protection assembly (APB). The assembly has the following purpose:

- Coupling the multi-purpose combustion tube with the other measuring gas paths
- Filtering solid particles, aerosols and liquids

The auto-protection assembly is attached to the furnace housing with a clamping mount.

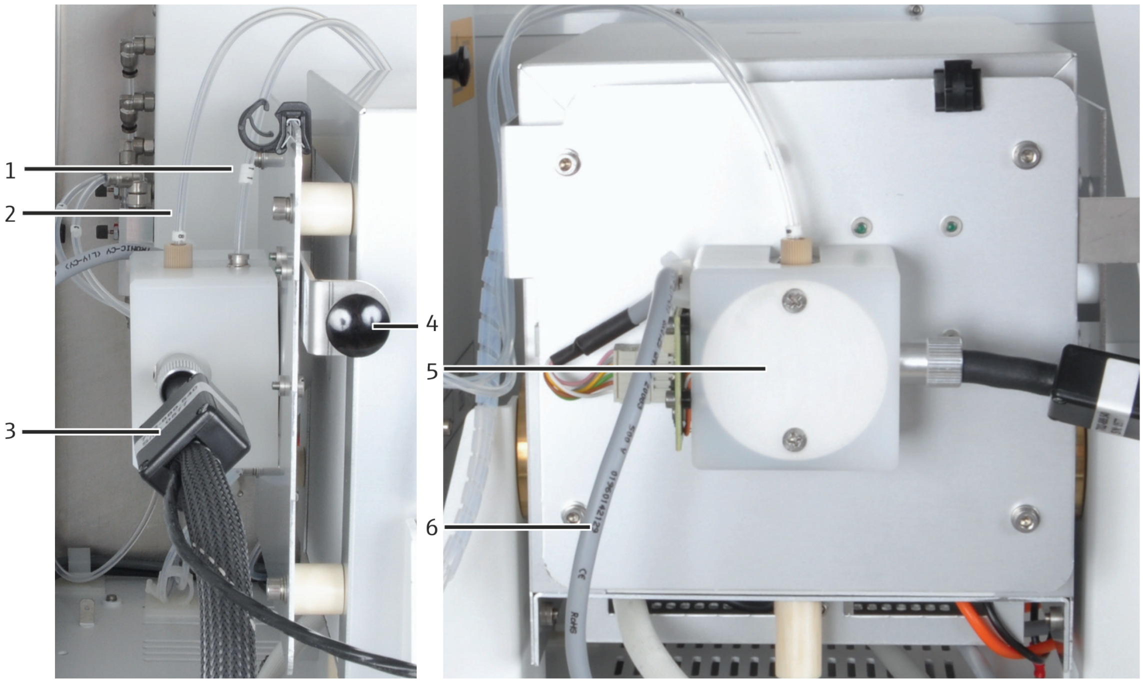


Fig. 16 Auto-protection assembly

1 Hose 11 (compressed argon gas for pneumatic seal)
2 Hose 8 (argon for safety purging)
3 Heated gas transfer line to the chlorine module
4 Knob of the clamping mount
5 Auto-protection assembly with integrated filter and pneumatic seal
6 Cable for electrical connection

Coupling with pneumatic seal

The coupling connects the combustion tube and the measuring gas transfer line. The combustion tube is inserted into the auto-protection assembly and sealed with a pneumatic seal. The pneumatic seal is operated with argon. The pneumatic seal is opened or closed with a toggle switch.

- Toggle switch down: Combustion tube sealed
- Toggle switch up: Combustion tube released (e.g. for dismantling)

Fig. 17 Toggle switch to lock/release combustion tube

Safety bypass

The argon safety bypass (2 in Fig. 1) on the auto-protection assembly prevents a vacuum from being created during cooling. This vacuum can draw sulfuric acid through the gas transfer line into the auto-protection assembly.

If the gas supply fails while the system is running, e.g., due to an emergency shutdown, or if the gas pressure drops due to a blockage while the system is running or cooling down, sulfuric acid may be blown back into the analysis system. Disconnect the heated transfer line from the sulfuric acid container immediately if the shielding gas supply is not ensured.

Filter

A replaceable filter has been integrated in the auto-protection assembly. The filter protects the downstream assemblies (drying units, detectors) in case of an incomplete combustion of carbon particles. The filter also retains condensed water and other aerosols. Only water vapor can pass through the filter.

3.5 Chlorine module multi X 2500

The chlorine module (Cl module) consists of the following main components:

- Wide-range coulometer for amperometric and potentiometric titration
- Stirrer/cooling block for the measuring cell (with automatic cell detection)
- Measuring cell with electrodes with gas inlet tube
- Intake pump
- Adsorption tube
- Sulfuric acid container for the operation with multi-purpose combustion tube

In the standard version, the intake pump draws the measuring gas from the multi X 2500 directly into the measuring cell of the chlorine module when working with an open combustion tube. The adsorption tube is introduced between the gas outlet of the measuring cell and the intake pump. The adsorption tube protects the intake pump against acetic acid vapors. Active carbon is used as adsorption agent.

In the chlorine module, the sulfuric acid container is only used when working with the optional multi-purpose combustion tube.

All components of the module which are operated or serviced by the user can be reached via the front door.

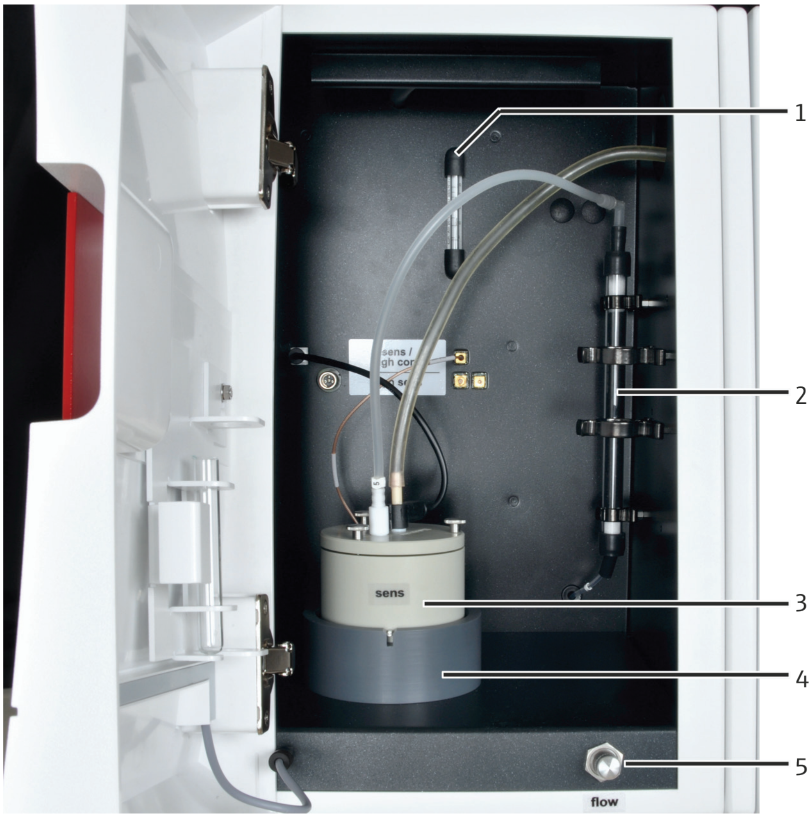


Fig. 18 **Cl module in the standard version**

1	Flow meter intake flow	4	Stirrer/cooling block
2	Adsorption tube	5	Flow control for intake flow
3	Measuring cell "sensitive" with combination electrode		

The intake flow is set to approximately 25 l/h when the device is commissioned.

The wide-range coulometer of the chlorine module has 3 operative ranges. A particular measuring cell is used for each operative range:

- "sensitive" (default)
- "high concentration" (optional)
- "high sensitive" (optional)

For further information on the measuring ranges of the measuring cells refer to "Technical data" on page 123. The measuring cells are automatically detected when they are inserted into the stirrer/cooling block.

When the chlorine 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 using the multiWin software.

3.5.1 Connections and interfaces

The green LED at front of the Cl module illuminates after the module has been switched on.

The mains switch, the device's fuse and the mains connection are located on the rear of the chlorine module. The RS 232 interface for connecting the multi X 2500 is also located on the rear.

The device's gas outlet is located in the bottom left corner of the rear panel. Depending on whether the device is operated as the standard version or as an optional version, a different outlet must be connected to the extraction:

- Outlet "exhaust" (2 in Fig. 17): Outlet of the intake pump with filter (standard version)
- Outlet "waste" (3 in Fig. 17): Outlet of the exhaust air hose (optional version)

The filter on the "exhaust" outlet creates a vortex in the exhaust gas stream which reduces the noise level.

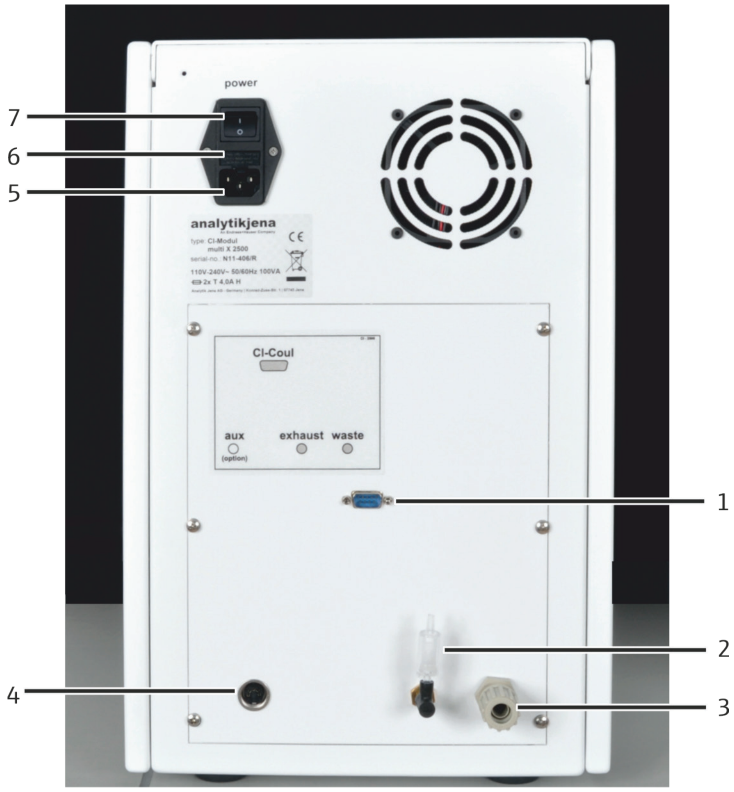


Fig. 19 Connections on the rear of the chlorine module

1 Interface to the basic device
2 Outlet intake pump ("exhaust")
3 Outlet exhaust air hose ("waste")
4 "Aux" Interface
5 Mains connection
6 Equipment fuse
7 Device switch

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

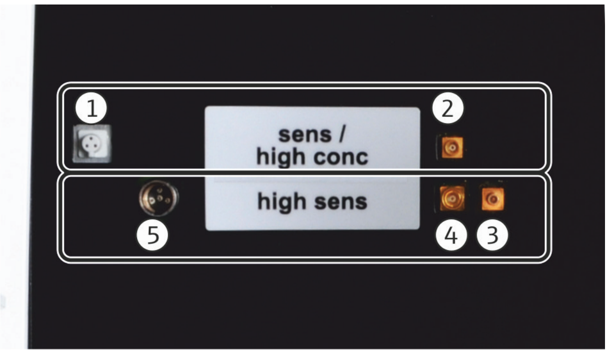


Fig. 20 **Terminal on the internal wall of the chlorine module**

For measuring cells "sensitive" and "high concentration"

1 Connection for the combined electrode

2 Connection for the measuring cell with silver anode

For measuring cell "high sensitive"

3 Connection platinum electrode

4 Connection silver electrode

5 Connection sensor electrode

3.5.2 Measuring cell "sensitive" (default)

Layout

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

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

The cell is sealed airtight with a cover and three knurled head screws. The two cover penetrations (holes) are used to mount the combined electrode and a suction olive. The marked opening is intended for the amperometric combined electrode. The unmarked opening is used for direct injection into the measuring cell or to connect the suction olive.

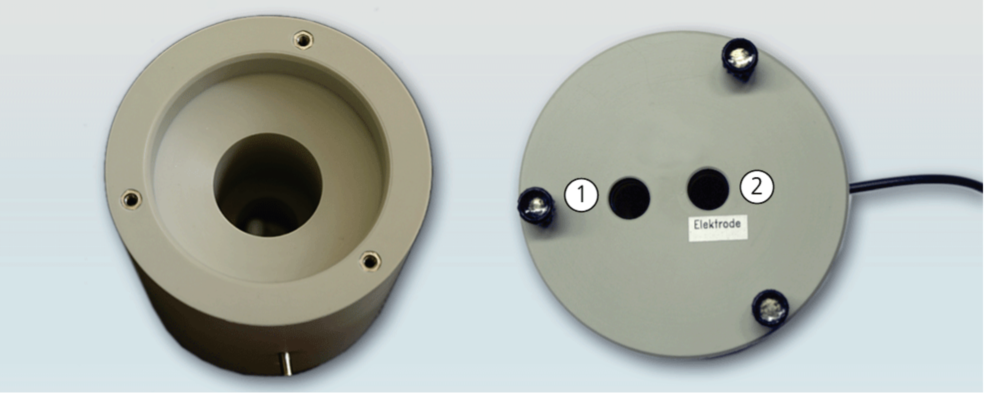


Fig. 21 **Measuring cell "sensitive" with lid**

1 Mount for olive-tip suction adapter
2 Mount for the combined electrode

Fig. 22 **Loaded measuring cell "sensitive"**

Combined electrode

The combined electrode is inserted into the measurement cells "sensitive" (default) and "high concentration" (option). 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 combination electrode is dried and stored in the storage container in the front door after the measurement.

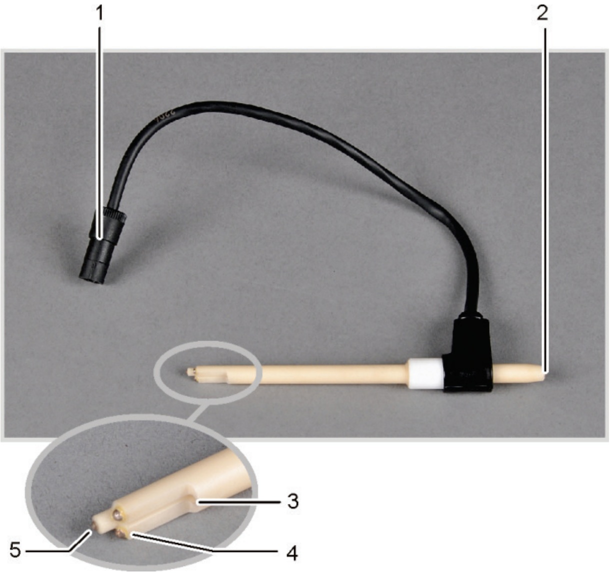


Fig. 23 **Electrode for "sensitive" & "high concentration" measuring cells**

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

3.5.3 Measuring cell "high concentration"

The measuring cell "high concentration" is offered as an option. It has the same function as the measuring cell "sensitive" but provides an elevated electrolyte volume. It can be used for chlorine contents between 10 and 1000 µg and is recommended in particular when determining TX in highly contaminated waste and polymer samples with high PVC content.

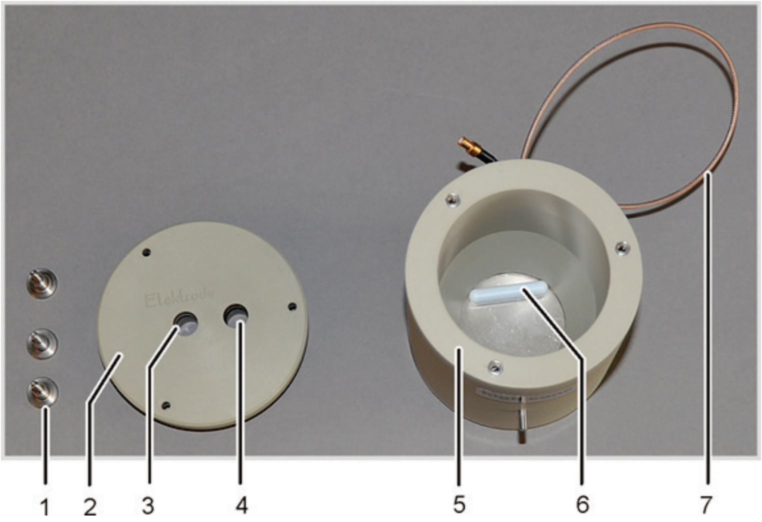


Fig. 24 **Measuring cell "high concentration"**

1 Knurled head screws
2 Cover
3 Opening for electrode (labeled)
4 Opening for olive-tip to connect aspiration hose or direct injection
5 Electrode space with silver anode
6 Magnetic stirring rod
7 Electrical connection of the measuring cell

Similar to the measuring cell "sensitive", this cell also uses a maintenance-free combined electrode.

3.5.4 Measuring cell "high sensitive"

Design

The optional measuring cell "high sensitive" is used for very low chlorine contents (between 0.01 and 10 µg Cl) in combination with the multi-purpose combustion tube. It is particularly recommended for EOX determinations. The measuring cell "high sensitive" cannot be used in combination with the open combustion tube (AOX and POX vertical).

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 one of the drilled holes is intended for an adapter which is connected to the extraction hose for the acetic acid vapors. The small unmarked opening is used for the direct injection in the cell. It is sealed with a plug when the gas from the combustion furnace is introduced.

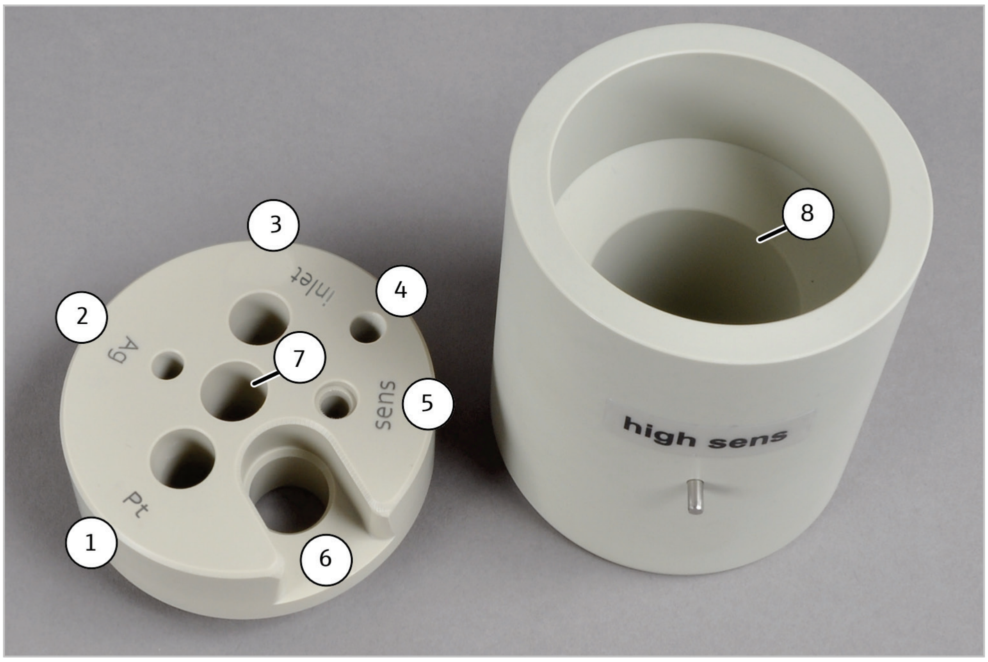


Fig. 25 **Measuring cell "high sensitive"**

1 Opening "Pt" (for the platinum electrode)
2 Opening "Ag" (for the silver electrode)
3 Opening "Gas" (for the gas inlet tube)
4 Opening for the direct injection
5 Opening "Sen" (for the sensor electrode)
6 Opening for the reference electrode
7 Opening for connection to extraction system
8 Base element of the measuring cell with magnetic stirring rod

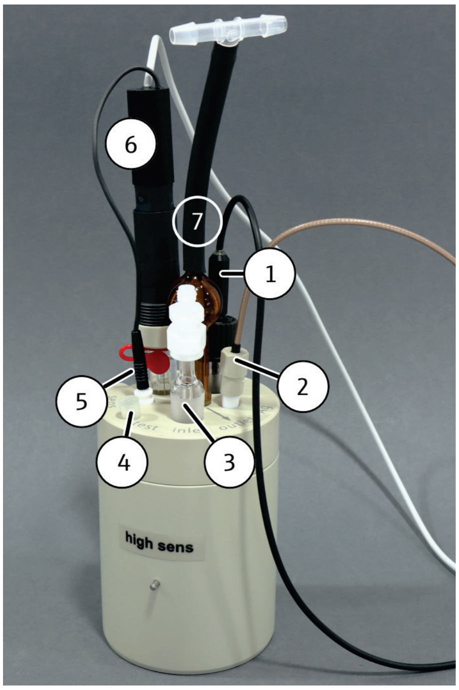


Fig. 26 **Loaded measuring cell "high sensitive"**

1 Platinum electrode with salt bridge
2 Silver electrode
3 Gas inlet tube with screw connection for measuring gas
4 Opening for the direct injection with plug
5 Sensor electrode
6 Reference electrode
7 Adapter to the extraction 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 refill opening and store the two electrodes in the measuring cell filled with electrolyte.

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.

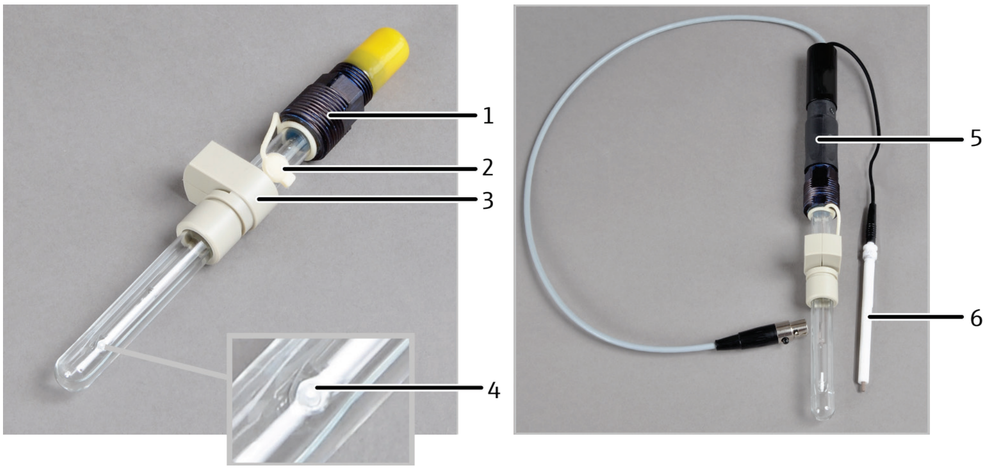


Fig. 27 Sensor electrode and reference electrode

1 Electrical connection
2 Refill opening for bridge electrolyte (with rubber plug)
3 Holder for reference electrode in the measuring cell cover
4 Diaphragm
5 Preamplifier
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.

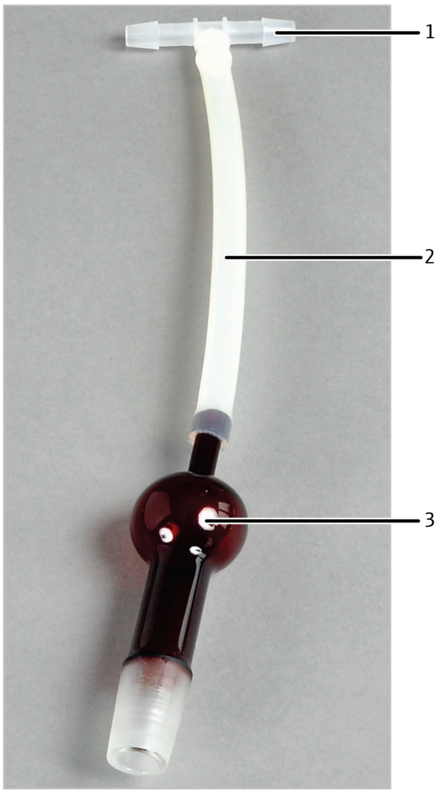


Fig. 28 Connection to extraction system

1	T piece	3	Adapter
2	Short hose		

Generator electrodes

A pair of generator electrodes consisting of a platinum cathode with salt bridge and a silver anode is used to produce the silver ions required for chemical precipitation.

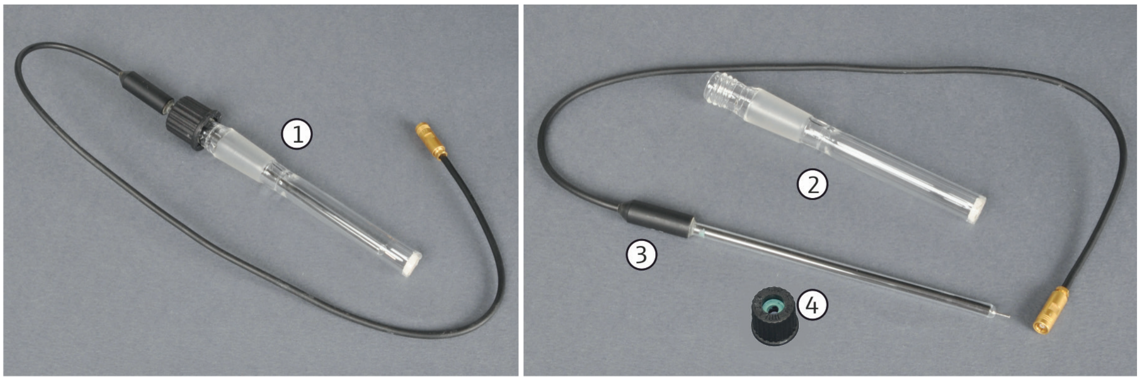


Fig. 29 Pt electrode with salt bridge (generator cathode)

1	Pt electrode with salt bridge, complete	3	Pt electrode
2	Salt bridge	4	Septum with opening (for attaching the Pt electrode to the salt bridge)

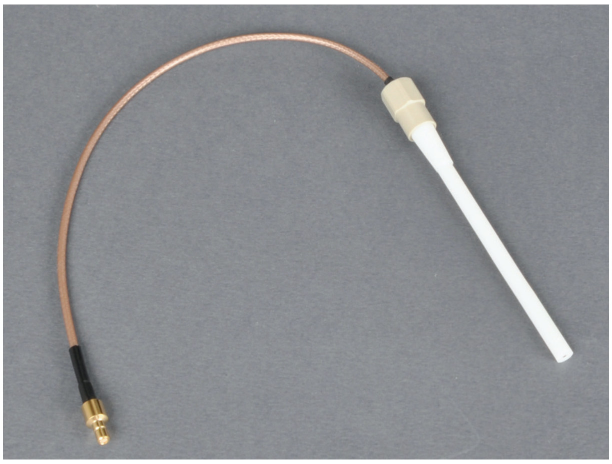


Fig. 30 Silver electrode (generator anode)

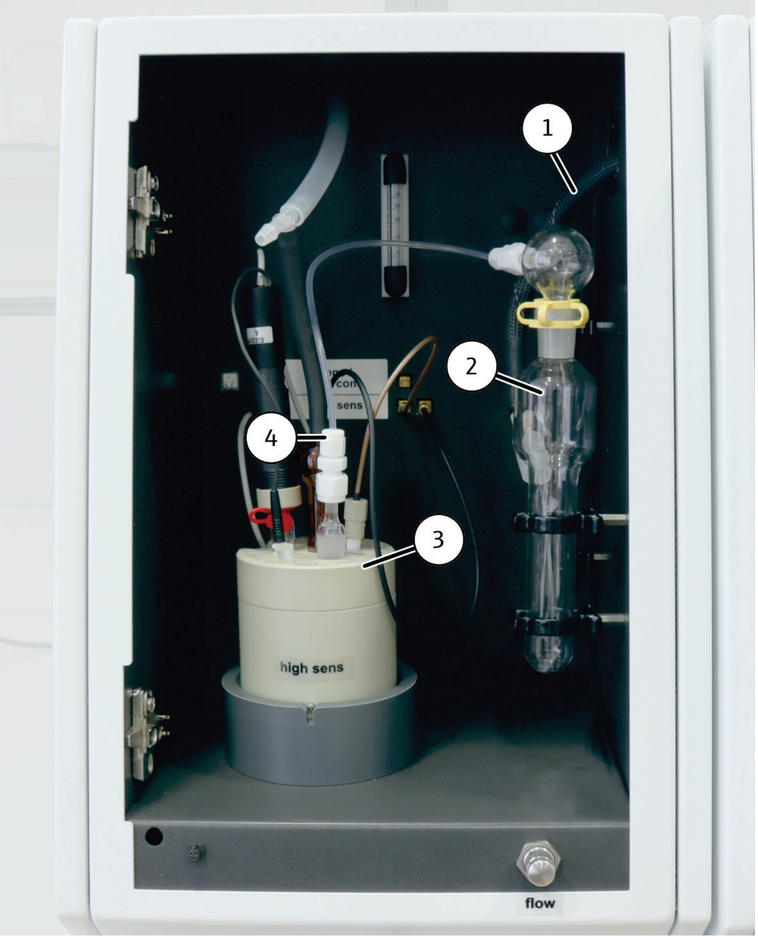


Fig. 31 Chlorine module with measuring cell "high sensitive"

1 Gas transfer line (from basic device)
2 Sulfuric acid container with safety attachment
3 Measuring cell "high sensitive"
4 Gas inlet to the measuring cell

3.5.5 Measuring gas transfer and measuring gas drying (optional versions)

Concentrated sulfuric acid is used for drying the measuring gas. There is a particular sulfuric acid container that is mounted to the chlorine module which can be used for working with the multi-purpose combustion tube. The measuring gas is transferred via a heated gas transfer line. It connects the combustion system of the multi X 2500 with the sulfuric acid container in the chlorine module. The hose of the gas transfer line is connected to the connector at the terminal of the sulfuric acid container using a banjo bolt with conical nipple. The other end of the gas transfer line is connected to the auto-protection assembly in the basic device.

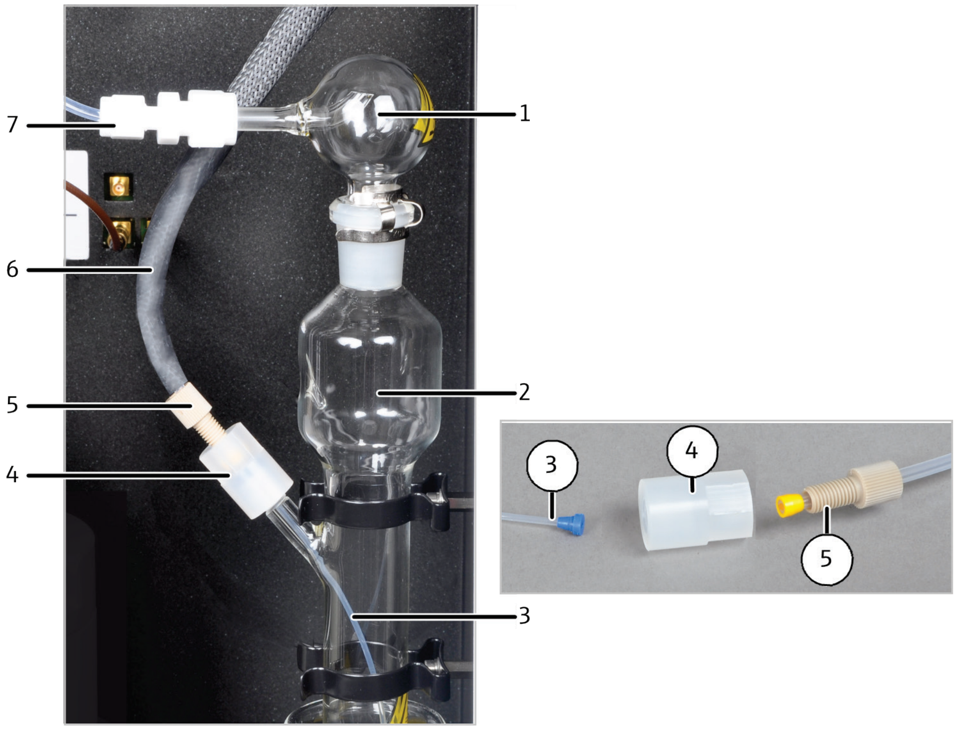


Fig. 32 **Sulfuric acid container in the chlorine module**

1 Safety attachment
2 Sulfuric acid container
3 Hose to introduce measuring gas
4 Connector
5 Banjo bolt
6 Measuring gas transfer to the measuring cell
7 Gas transfer line to measuring cell

The combined electrode is used to introduce the measuring gas to the measuring cells "sensitive" and "high concentration". A ground glass tube with and Teflon screw connection for connecting the measuring gas hose is used to introduce the gas to the measuring cell "high sensitive".

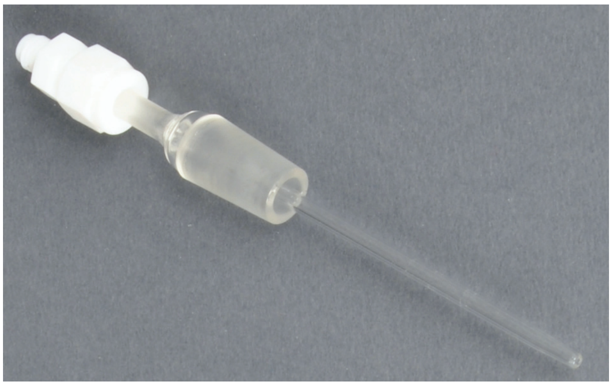


Fig. 33 **Gas inlet tube for the measuring cell "high sensitive"**

Measuring cell "high sensitive" Measuring cells "sensitive" and "high concentration"

Fig. 34 Introducing gas to the measuring cell (multi-purpose combustion tube)

3.6 Autosampler / sample introduction systems

The following autosamplers and sample introduction systems are available for the multi X 2500:

- Autosampler autoX 36 / autoX 36d
- Autosampler autoX 112
- Automatic boat feeder ABD
- Manual boat feeder MBD
- Auto-injector

The autosamplers autoX 36 and autoX 36d were specifically conceived for the multi X 2500. The autosamplers are described in this operating manual.

The manual boat feeder (MBD) is also described in this manual. When using any other sample introduction module, refer to the respective operating instructions.

3.6.1 autoX 36 / autoX 36d

The autosamplers autoX 36 and autoX 36d are used for small AOX sample series in vertical operating mode. They can transfer 36 samples into the open combustion tube.

- **autoX 36**: Autosampler used to automatically introduce 36 quartz containers (18 x 6 mm, 18 x 8 mm) with activated carbon which has been enriched using the column or the batch method.

- autoX 36d: Autosampler with ejection function for direct dosing of activated carbon from 36 columns (18 x 6 mm). It can be employed for samples which were enriched using the column method.

Both autosamplers are connected to the multi X 2500 by means of the interface cable. The multiWin control software automatically detects autosamplers and sample racks and controls the introduction of samples. A cover ensures that cross-contamination and environmental influences can be excluded. On the autoX 36d autosampler the cover is also used to push down the quartz containers during the ejection process. The floating arrangement allows the autosampler to be inserted in the right position of the oxygen sluice in the cutout of the basic device.

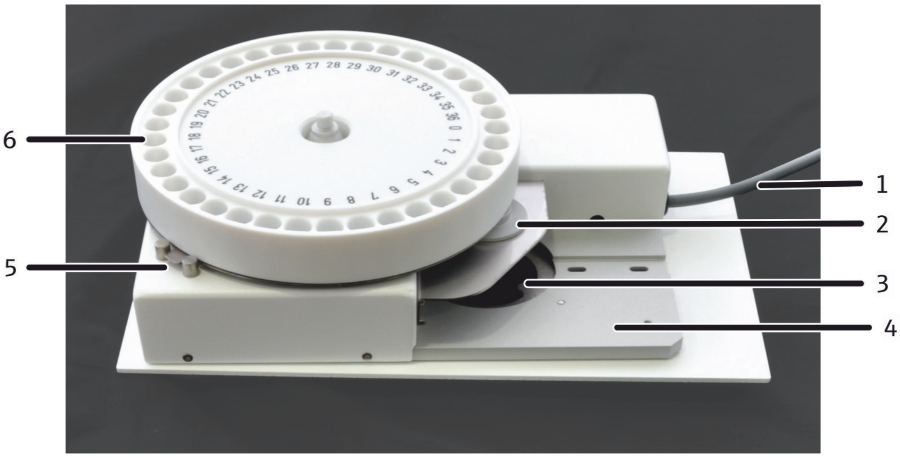


Fig. 35 **Autosampler autoX 36**

1 Interface cable
2 Drop position
3 Cutout for the oxygen sluice
4 Floating bearing arrangement
5 Lug for correct positioning of the drop position
6 Rack

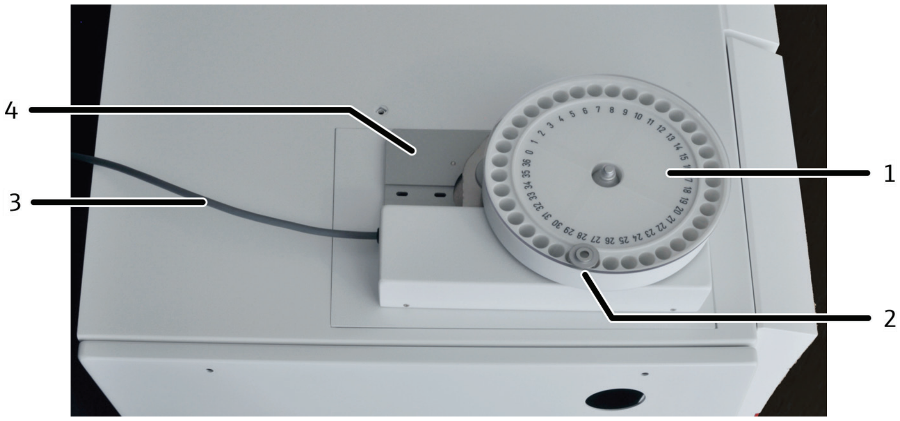


Fig. 36 **autoX 36 (stacked on the multi X 2500)**

1 Autosampler autoX 36
2 Cover with ejection function
3 Interface cable
4 Mount in the cutout of the multi X 2500

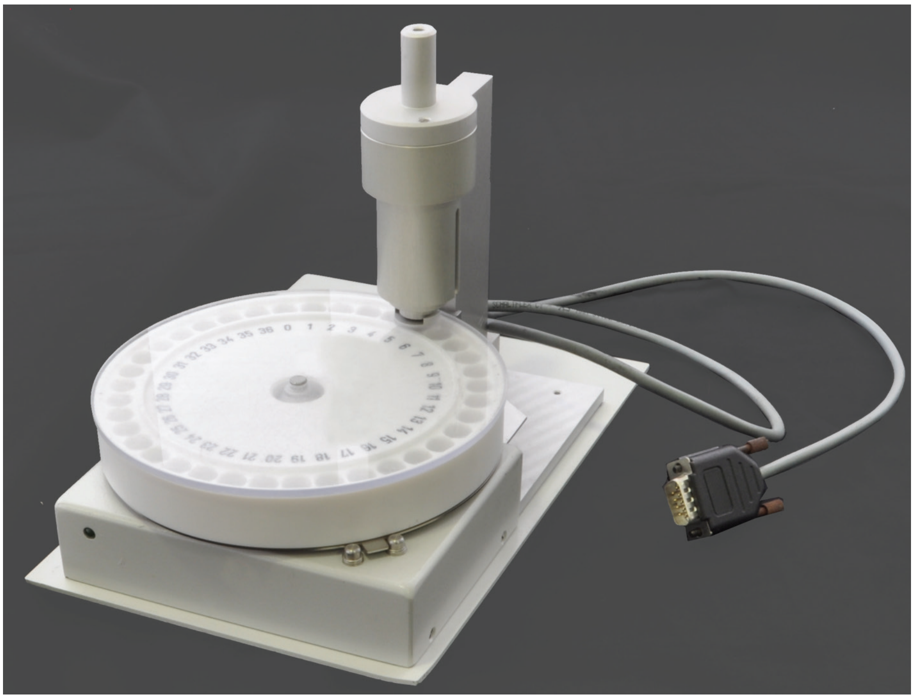


Fig. 37 autoX 36d with ejection position

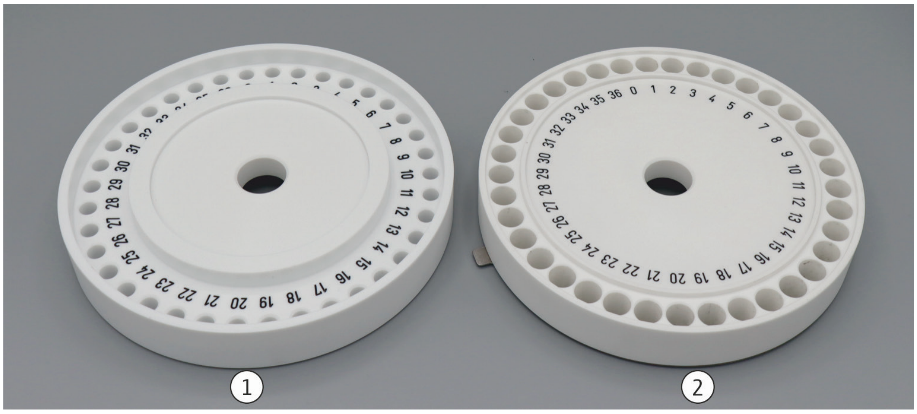


Fig. 38 Sample racks

1 Sample rack for autoX 36d
2 Sample rack for autoX 36

3.6.2 Manual boat feed (MBD)

The sample feeding module MBD (manual boat drive) is a sample introduction system for AOX determination and the analysis of inert solids in horizontal operating mode. The MBD is used for transporting the quartz boats (40 mm x 9 mm) into the multi-purpose combustion tube. The quartz boats can be loaded with quartz containers with activated carbon (column method) or with filters with filter cakes (batch method). For safety reasons the analysis of liquid samples is not permitted.

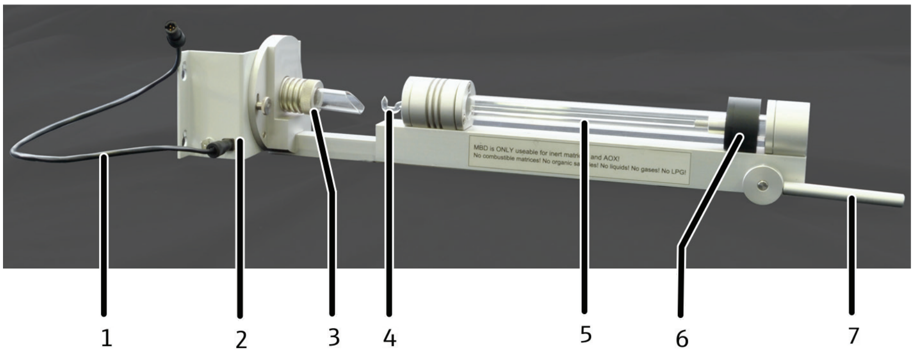


Fig. 39 Manual boat feed (MBD)

1 Connection cable
2 Bracket
3 Connection piece with seal
4 Hook
5 Guide tube
6 Handle for sliding the hook
7 Lever for opening and closing the sluice

The guide tube includes a hook with magnetic coupling which is used to move the loaded quartz boat. Opening and closing the sluice and transferring the samples into the combustion tube are performed manually.

The MBD is hooked to the three fastening screws on the combustion furnace by its bracket. MBD and combustion tube are connected using a connection piece which is screwed onto the combustion tube. It ensures that the connection is gas tight.

The MBD is connected to the interface in the device interior (3 in Fig. 5 on page 20) using the connection cable. The MBD is then identified by the multiWin software. Argon is used as carrier and cooling gas. For this purpose, the MBD is connected to the "OUT ABD" gas outlet of the multi X 2500 using the connection hose (6 in Fig. 7 on page 21).

3.7 Pre-combustion adapter

The pre-combustion adapter is an optional accessory for samples which are wet and which must be dried before the actual combustion process. It is provided as a particular option for examining sewage sludge.

The pre-combustion adapter can be used together with the open combustion tube in vertical operating mode (standard version).

The pre-combustion adapter can be used for drying frit containers with wet filter cakes like those produced when using the batch method. The batch method is predominantly used for the AOX determination from turbid samples, sludge and sediments (→ "Sample preparation" on page 52).

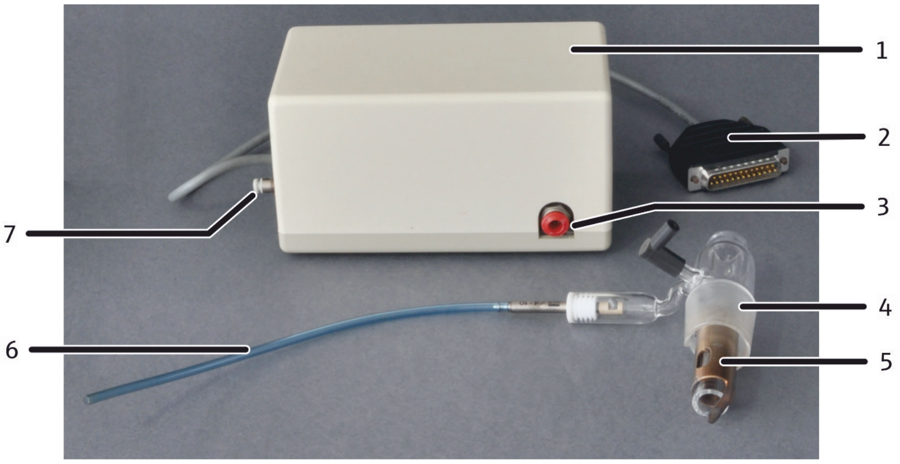


Fig. 40 Pre-combustion adapter

1	Pneumatic box	5	Clamping device for the sample
2	Interface cable	6	Pneumatic unit with gas hose
3	Connection to oxygen supply	7	Gas connection on the multi X 2500
4	Pre-combustion adapter		

The pre-combustion adapter is attached to the open combustion tube instead of the oxygen sluice. In addition to the oxygen connection it has a pneumatic unit which is used to move the clamping device for the sample downward. The pre-combustion adapter is connected to the multi X 2500 via the pneumatic box. It is then possible to use the multiWin control software to determine for how long the sample is supposed to be pre-dried in the upper part of the combustion tube clamped in between the glass tube and the clamping device. After the preset time has elapsed, a gas blast pushes the clamping device downward. The sample drops into the hot combustion tube where it is combusted in the oxygen stream.

3.8 Mini Scrubber Kit

The Mini Scrubber Kit is an air purification module that is intended as an accessory for chlorine determination with a "high sensitive" measuring cell. During operation of this measuring cell, acetic waste air is produced. The optional accessory cleans this waste air and the analyzer can thus be operated irrespective of being connected to a laboratory exhaust system.

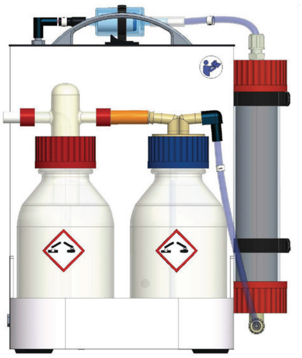


Fig. 41 Mini Scrubber Kit

4 Installation and commissioning

4.1 Installation conditions

Ambient conditions

See "Technical data" on page 123 for information on the ambient conditions of the installation location. It may be necessary to control the temperature in the room.

Laboratory conditions

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
- Smoking must be prohibited in the room where the multi X 2500 is operated

Requirements with regard to the installation location

The site for installing the multi X 2500 must meet the following requirements:

- Free from caustic vapors in close proximity of the multi X 2500
  Connections and assemblies of the device may corrode.
- 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
- Keep a safety distance of at least 20 cm from the rear of the device to other equipment or walls

4.2 Energy supply

Caution

The multi X 2500 must only be connected to a power outlet which is properly grounded in accordance with the voltage specifications on the type plate!

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

Electrical connection data, see "Technical data", p. 123 ff.

4.3 Gas supply

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

The connection hoses with outer diameter 6 mm and inner diameter 4 mm are included with the delivery. The length is 2 m. If other lengths are preferred, please contact the Analytik Jena customer service. Required gases, see section "Technical data", p. 123 ff.

4.4 Space requirement and device layout

The space requirements are based on the system modules required for the measuring task. In addition to that, the setup requires space for a PC, a monitor and a printer.

Dimensions

Device	Dimensions (width x height x depth) in cm
Basic device multi X 2500	51 x 47 x 56
Cl module or TOC module (each)	30 x 47 x 56
autoX 36: autoX 36d:	14.5 x 6.5 x 24 14.5 x 18 x 24
autoX 112:	51 x 50 x 40
ABD	52 x 21 x 50

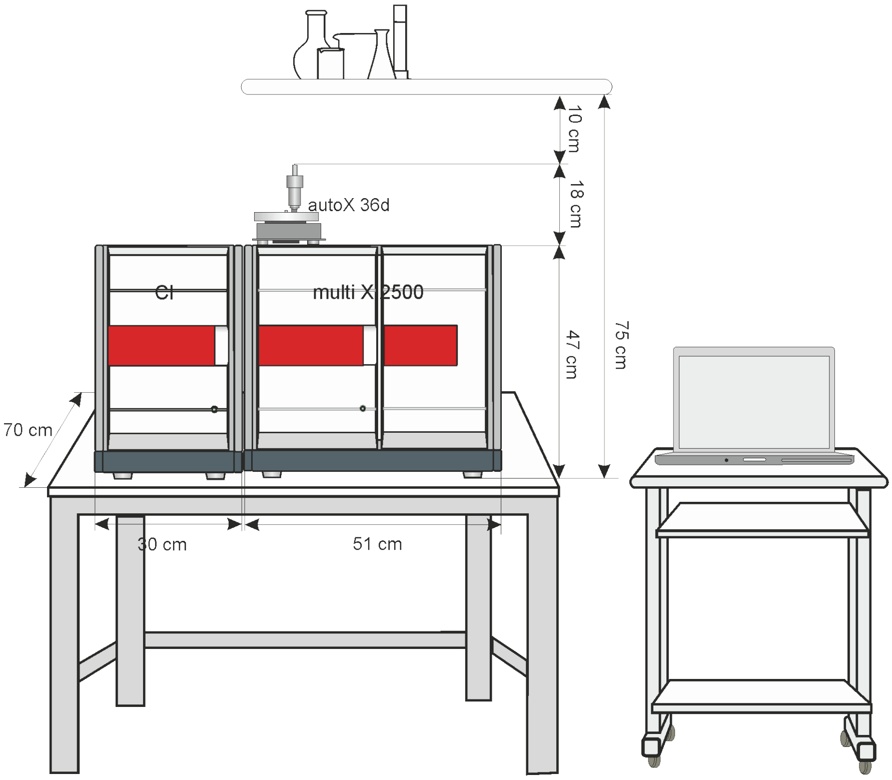


Fig. 42 Vertical operating mode with autoX 36d

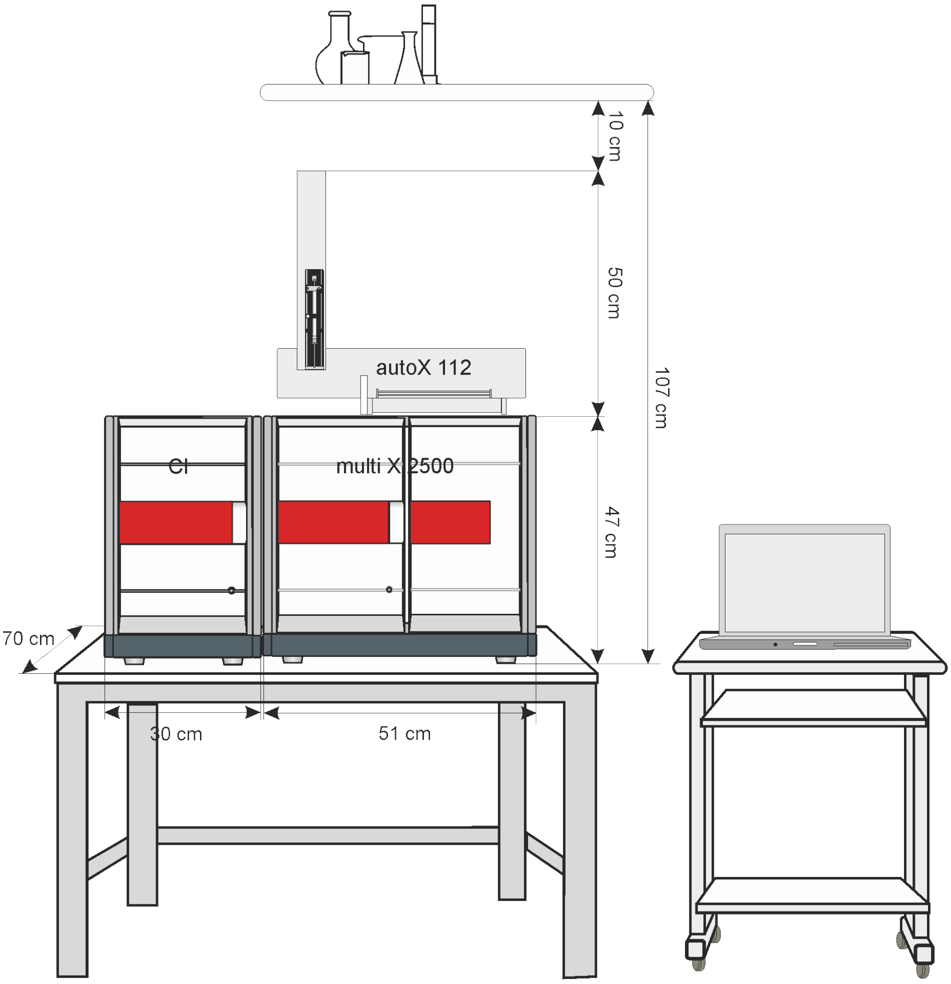


Fig. 43 Vertical operating mode with autoX 112

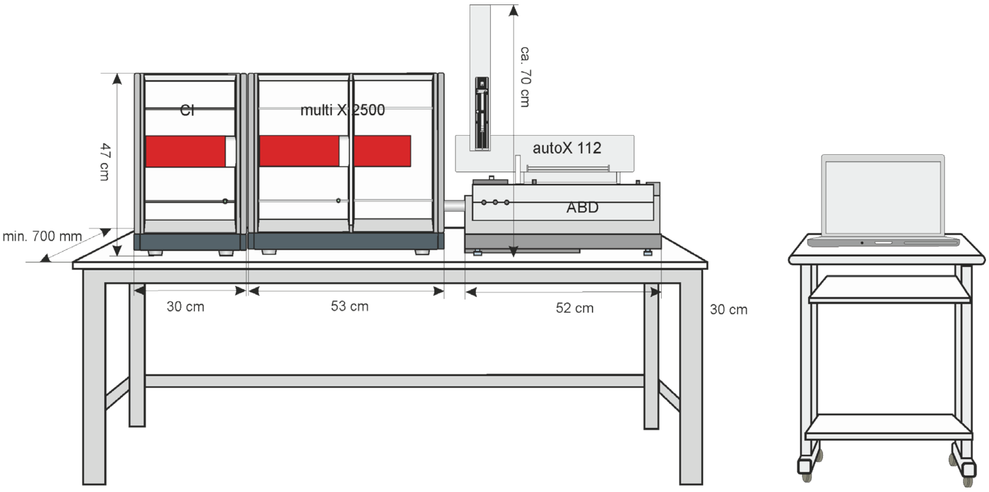


Fig. 44 Horizontal operating mode with ABD and autoX 112

4.5 Setting up and connecting the analyzer

CAUTION

The multi X 2500 must only be set up, assembled and installed by the customer service department of Analytik Jena or trained specialist personnel authorized by Analytik Jena!

Any unauthorized intervention on the multi X 2500 can endanger the user and the operational safety of the equipment and limits or completely invalidates any warranty claims.

NOTICE

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

The basic module is unpacked and assembled by the customer service department of Analytik Jena or authorized and trained specialist personnel.

Check when unpacking the device for completeness and soundness of the delivery in accordance with the packing list included. After assembly the customer service tests the functionality of the multi X 2500 and other system components and documents the test.

Connecting the multi X 2500

NOTICE

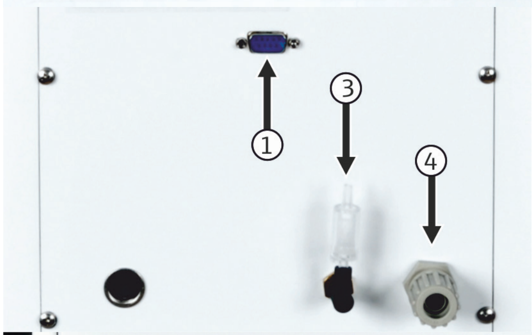
Always switch off all components of the multi X 2500 before electrically connecting them! Connecting or disconnecting electrical contacts may damage the sensitive electronic components of the multi X 2500 and detector modules.

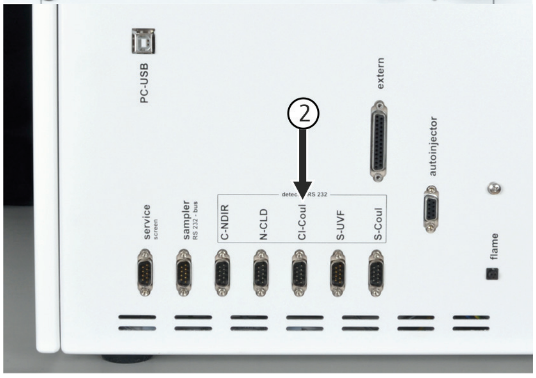
NOTICE

Condensed water and temperature differences may damage the electronic components of the multi X 2500.

Allow the multi X 2500 to acclimatize in the operating room for one hour before putting the device into service after transport and storage.

1. Connect the mains cables to the mains connections of the multi X 2500 and the chlorine module. Plug in the mains cable to the mains power outlet.

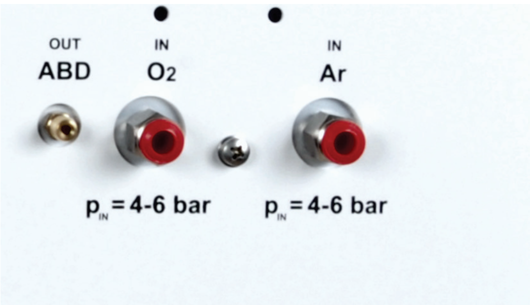




2. Connect the multi X 2500 and the chlorine module using the serial cable.
   - **Cl-Coul** interface on the rear of the chlorine module (1)
   - **Cl-Coul** interface on the rear of the basic device (2)

Standard version (AOX vertical):

3. Connect the exhaust air hose to the outlet "exhaust" of the chlorine module (3). Connect the hose to an extraction system or place it into an exhaust vent.
4. Connect the provided gas hose to the oxygen supply of the laboratory and the gas connection **O_2 IN** on the rear of the device.



Optional versions (AOX horizontal, EOX, POX, TX):

5. Connect the exhaust air hose to the outlet "waste" of the chlorine module (4). Connect the hose to an extraction system or place it into an exhaust vent.
6. Connect the provided gas hose to the oxygen supply of the laboratory and the gas connection **O_2 IN** on the rear of the device.
7. Connect the provided gas hose to the argon supply of the laboratory and the gas connection **Ar IN** on the rear of the device.

The outlet "exhaust" on the basic device does not need to be connected to the extraction system because this outlet is not used to expel exhaust air to the surrounding air.

4.6 Connecting additional system components

The proper connection of additional system components to the multi X 2500 basic device is described in the user manual of the respective system component:

- Sample feeder autoX 112:
- Auto-injector
- Automatic boat feeder (ABD)
- Manual boat feeder (MBD)
- TOC module

5 Operation

5.1 Switching on the analyzer

Before switching on check the following:

- The carrier gases are connected with a primary pressure of 4 to 6 bar.
- A combustion tube has been inserted in the furnace.
- The gas supply hoses are connected.
- The chlorine module is connected.
- The autosampler (optional) is installed.

1. The analyzer and the chlorine module are switched on.
   - ✓ The devices are booted. The status LEDs on the front panels light up in green after approximately 30 seconds.
2. Switch on the PC.
3. Start the multiWin control and analysis software. Log in with user name and password.
4. Initialize the analysis system by clicking on [INITIALIZE ANALYZER].
   - ✓ The initialization and automatic detection of all connected components will be executed.

Warm-up and run-in phases

After logging in, the components are initialized and polled. The current values are displayed in the status window:

- Furnace temperature: The preheating time is approximately 30 to 40 minutes, depending on the target temperature.
- Wide-range coulometer: approx. 10 min warm-up time.
- NDIR detector (only for TOC determination): This assembly has a warm-up time of approx. 15 min. after the TOC module is switched on.

In the status window the displays for the components that are not yet ready for operation are red during this time.

5.2 Switching off the analyzer

WARNING

Concentrated sulfuric acid can cause severe chemical burns! Wear protective clothing when working at the sulfuric acid container. Observe all specifications in the safety data sheet!

Standard version

1. Exit the multiWin control and analysis software. Allow the device to cool down.

   ATTENTION! Wait for a moment before switching off the basic device and the chlorine module. Otherwise, the device may overheat when the ventilation is switched off with the device.

2. Use the device switch to switch off the basic device and the chlorine module.
3. Remove the electrode from the measuring cell and store it in accordance with the applicable regulations (→ "Clean and store the combined electrode", p. 92).
4. Empty the measuring cell and clean it (→ "Servicing the measuring cell", p. 91).
5. After the device has cooled down, remove the sulfuric acid container from the basic device and clean it.

Optional versions

Note

Do not cut the gas supply before the sulfuric acid has been removed or the device has cooled down. A vacuum may build up when the device cools down which may draw sulfuric acid into the gas transfer line. As long as the gas supply is open, the argon safety bypass on the auto-protection assembly prevents this from happening.

If the gas supply fails while the system is running, e.g., due to an emergency shutdown, or if the gas pressure drops due to a blockage while the system is running or cooling down, sulfuric acid may be blown back into the analysis system. Disconnect the heated transfer line from the sulfuric acid container immediately if the shielding gas supply is not ensured.

1. Exit the multiWin control and analysis software.
2. Disconnect the gas transfer pipe from the sulfuric acid container in the chlorine module. Remove the sulfuric acid from the sulfuric acid container. (→ "Changing the sulfuric acid and cleaning the sulfuric acid container" p. 101).

   WARNING! Sulfuric acid can cause severe chemical burns. The grinding of the sulfuric acid container and the hose which is immersed in the sulfuric acid container may contain residual 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.

3. Allow the analyzer to cool down. After that, switch off the multi X 2500 and the chlorine module using the mains switch. Cut the gas supply.
4. Remove the electrode from the measuring cell and store it in accordance with the applicable regulations (→ "Cleaning and storing the electrodes", p. 103).
5. Empty the measuring cell and clean it (→ "Measuring cell maintenance", p. 103).

5.3 Restarting the analyzer after an emergency shutdown

WARNING

Concentrated sulfuric acid can cause severe chemical burns! Wear protective clothing when working at the sulfuric acid container and the gas transfer line! Observe all specifications in the safety data sheet!

CAUTION

Risk of burns from the hot furnace and from the heated gas transfer line. Allow the device to cool before recommissioning.

If the multi X 2500 is operated with an optional multi-purpose combustion tube, there may be residual sulfuric acid in the gas transfer line after an emergency shutdown. Take particular care when checking the gas transfer line!

Observe the following instructions when putting the analyzer back into service:

1. Remove the gas transfer line from the sulfuric acid container in the chlorine module and remove the sulfuric acid from the sulfuric acid container (→ "Changing the sulfuric acid and cleaning the sulfuric acid container", p. 101).
2. Disconnect the gas transfer line inside the basic device from the auto-protection assembly and remove the connector of the heating cable from the socket.
3. Carefully remove the gas transfer line from the basic device and check it for contamination with sulfuric acid.
4. 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).
5. Refit the gas transfer line:

   To do this, connect the gas transfer line to the auto-protection assembly and plug the connector of the heating cable into the socket.

   Route the gas transfer line through the side panel to the chlorine module and connect it to the sulfuric acid container (→ "Preparing the chlorine module", p. 68).
6. Refill the sulfuric acid container with sulfuric acid and reinstall it on the chlorine module.

6 AOX determination

The samples for an AOX determination are prepared in accordance with column or batch method stipulated in the respective standard. The loaded activated carbon is combusted in the oxygen stream. During this process, the organically bound halogens are transformed into hydrogen halides whose mass concentration is determined as chloride.

The multi X 2500 can perform the AOX determination in both vertical and horizontal operating mode. The default version in vertical operating mode is recommended. The technical equipment for the standard version is simple, it requires little space and has low operating costs. The duration of the analysis is relatively short.

Working in horizontal mode requires an extension kit and an automatic boat feeder (ABD) or manual feed (MBD) for moving the quartz boats with the loaded activated carbon into the combustion tube.

6.1 Sample preparation

6.1.1 Sample preparation for AOX determination from waters

Basic information

Please observe the following:

- The AOX method can be used to determine AOX contents of more than 10 µg/l directly.
- The DOC content should be less than 100 mg/l; otherwise, the sample must be diluted (especially in the presence of alcohols, aromatics or carboxylic acids).
- The content of inorganic chloride or bromide should be smaller than 1 g/l. Otherwise, the sample must be diluted or replaced by a solid phase extraction for preparing the sample (SPE-AOX method), for example.
- If water samples (e.g. Cl_2, I_2 or ClO_3) are mixed with Na_2SO_3 for sample preparation, it is important to avoid an excess of Na_2SO_3.
- If the sample contains living cells, the sample must rest for eight hours after acidification before further analytic processing.
- Samples must be examined as soon as possible. Where this is not possible, the samples must be stored at 4 °C.

Sample preparation

Preparing the nitrate stock solution

WARNING

Concentrated nitric acid can cause severe chemical burns! Wear protective clothing when preparing the nitrate stock solution! Observe all specifications in the safety data sheet!

1. Dilute 17 g of $NaNO_3$ in a 1-liter volumetric flask with water.
2. Add 25 ml of HNO_3 (conc.).
3. Top up with water to 1000 ml.

Preparing the sample:

1. Make sure that the water sample has room temperature before initiating the adsorption.
2. Add 5 ml of nitrate stock solution to 100 ml of the sample.
3. Set the pH-value of the solution to $pH < 2$ using HNO_3.
   - ✓ The prepared solution is now ready for adsorption.

Column Method

The sample is adsorbed into quartz containers filled with 50 mg of activated carbon (AOX carbon). The activated carbon is fixed with ceramic glass wool plugs.

For adsorption according to the column method, the automatic sample preparation modules APU 28 connect S, APU 28 connect SPE and their flexi versions, APU*sim* or AFU 3 (with the column method kit) can be used.

All devices and tools required for the preparation and execution of the AOX adsorption are delivered with the sample preparation module.

Batch method

The batch method requires more effort than the column method, but is often preferred for the adsorption from turbid samples. Note that high chloride concentrations result in a higher distortion than they produce with the column method.

1. Add 100 ml of the sample, 5 ml of nitrate stock solution and 50 mg AOX carbon to a 250-ml Erlenmeyer flask. Use a plug to seal the flask.

   The AJ filling device is recommended for metering the AOX carbon.
2. Shake the suspension for 1 hour.
3. Use a membrane filter apparatus to separate water/activated carbon or water/activated carbon/sludge with a chloride-free polycarbonate membrane filter (0.45 µm, 25 mm diameter).
4. Add 50 ml of the nitrate stock solution to a 1-liter volumetric flask and top up the flask with water (nitrate wash solution).
5. After passing the AOX carbon, use at least 25 ml of the nitrate wash solution to wash the system in portions until it is free of chloride.

   When filtering, make sure that as little laboratory air as possible is drawn in and passed through the AOX carbon.
6. For the combustion in vertical operating mode, push the filter including the wet filter cake into a chloride-free calcined frit container (18 x 6 mm or 18 x 8 mm) and fix it using some ceramic glass wool.

   For the combustion in horizontal operating mode (multi-purpose combustion tube), the filter and the wet filter cake are pushed into a quartz glass boat with holding-down clamp.

AFU 3

As an alternative to the membrane filtration, the separation of water/activated carbon or water/activated carbon/sludge can be effected using the automatic filtration unit AFU 3. For this process, the activated carbon or the mixture of activated carbon and sludge is pressed into a frit container (18 x 8 mm) using overpressure of up to 2 bar.

The samples can then be combusted and analyzed in vertical operating mode (with open combustion tube).

6.1.2 Sample preparation for AOX determination from sludge and sediments

Enrichment and filtration are generally effected as described for the batch method (→ "Sample preparation for AOX determination from waters", p. 52). Depending on the expected AOX content, add 10 to 100 mg of the sample (e.g. sewage sludge) and approximately 20 mg of AOX carbon to a 25-ml Erlenmeyer flask containing 10 ml of nitrate stock solution and shake the mixture for 1 hour. After that, the samples are filtered using a membrane filtration apparatus or the AFU 3, washed with nitrate wash solution and combusted.

6.2 AOX determination in vertical operating mode (recommended)

Sample preparation	Column and batch method
Operating mode:	Vertical:
Measuring cell	"sensitive"
Measuring range	1 to 100 µg Cl
Electrolyte volume	20 ml
Combustion tube	Open combustion tube with pre-combustion adapter (batch method) as an option
manual sample introduction	Without accessories
automatic sample introduction	One of the following samplers: autoX 36 (for column method or batch method) autoX 36d (column method) autoX 112 with AOX rack (column method)

For AOX determination in vertical operating mode the analyzer is operated using the default configuration. It is equipped with the open combustion tube and the measuring cell "sensitive".

There is the option to equip the analyzer with a pre-combustion adapter for analyzing sewage sludge and sediments.

When using the autosampler autoX 112, observe the particular instructions in the respective user manual!

6.2.1 Preparing the analyzer (standard version)

The preparation of the analyzer includes the following steps:

- Insert the open combustion tube
- Insert the sulfuric acid container (vertically)
- Prepare the chlorine module

The preparation of the electrolyte solution and the end point routine for the measuring cell are described in the subsequent chapter (→ "Preparing the measuring cell", p. 59).

Inserting the open combustion tube

CAUTION

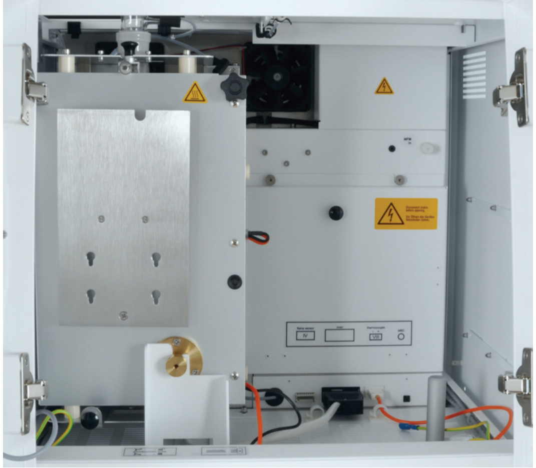
Risk of burns! Only install the combustion tube in the cold state!

When working with quartz wool avoid the creation of dust! Inhaled dust might cause irritation to respiratory tracts.

NOTICE

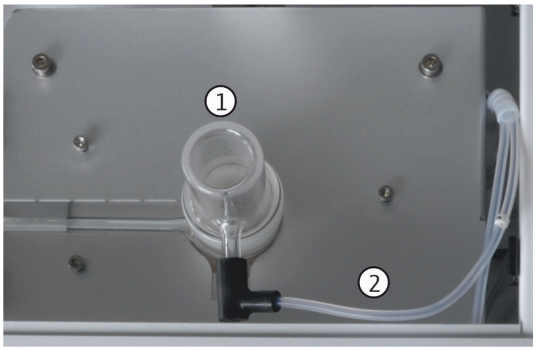
Alkaline salts (hand perspiration) cause crystallizations in the quartz glass when heating the combustion furnace which reduce the service life of the combustion tube.

Do not touch the combustion tube with your hands. Wear protective gloves. If necessary, clean the combustion tube externally before installing it in the combustion furnace (e.g. by wiping it with moist cellulose).



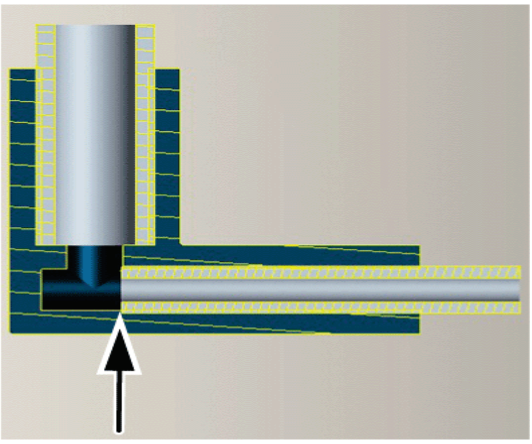
1. Exit the multiWin control software. Wait until the basic device has cooled down.
2. Switch off the analyzer and the chlorine module using the mains switch and pull the mains plug from the power outlet. Cut the gas supply.
3. Remove the top cover and open the front doors.
4. If not yet done, tilt the combustion furnace into the vertical installation position.

5. Carefully push a quartz wool plug into the open combustion tube until it reaches the bottom.



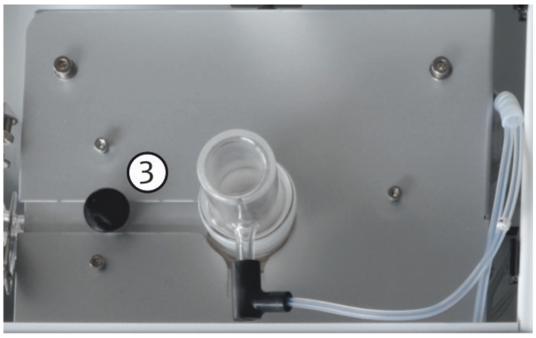
6. Insert the combustion tube into the combustion furnace. When doing this, make sure that the connections on the combustion tube fit into the recesses on the furnace.
7. Attach the oxygen sluice (1) to the top of the combustion tube.

   Connect the angled adapter (2) with the oxygen hose (hose no. 3) to the oxygen sluice.



NOTICE! Do not push the oxygen hose and the connector beyond the branch length of the angled adapter on the sluice (see arrow). Only then, the gas will be able to flow freely.

✓ The open combustion tube is mounted and can be connected to the sulfuric acid container.



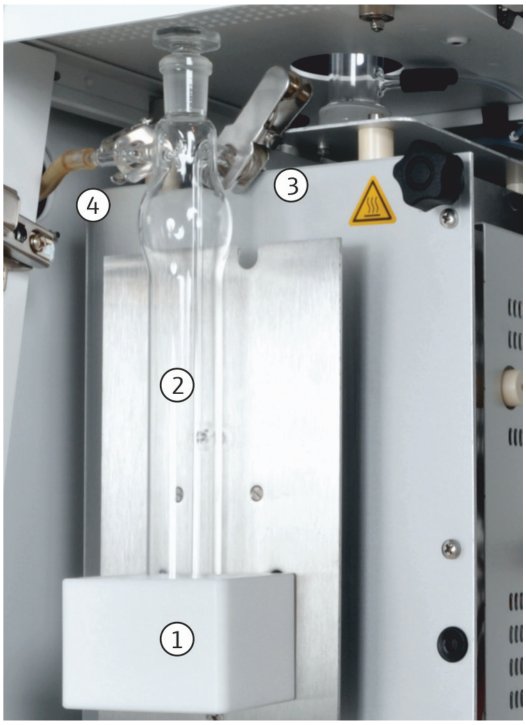
8. Mount the heat bridge in the cutout for the furnace above the gas outlet of the combustion tube.

Insert the sulfuric acid container (vertically):

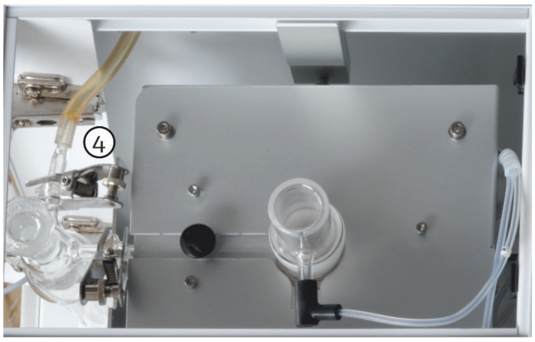
WARNING

Concentrated sulfuric acid can cause severe chemical burns! Wear appropriate protective clothing when working at the sulfuric acid container. Observe all specifications in the safety data sheet!

In the standard configuration, the sulfuric acid container is attached to the furnace of the multi X 2500 and directly connected to the open combustion tube via a spherical joint.



1. Attach the drip pan (1) to the combustion furnace.
2. Fill the sulfuric acid container (vertical, 2) with 40 ml of concentrated sulfuric acid.
3. Insert the plug to the sulfuric acid container.
4. Carefully place the sulfuric acid container in the drip pan of the combustion furnace.
5. Connect the sulfuric acid container to the open combustion tube using the forked clamp (3).

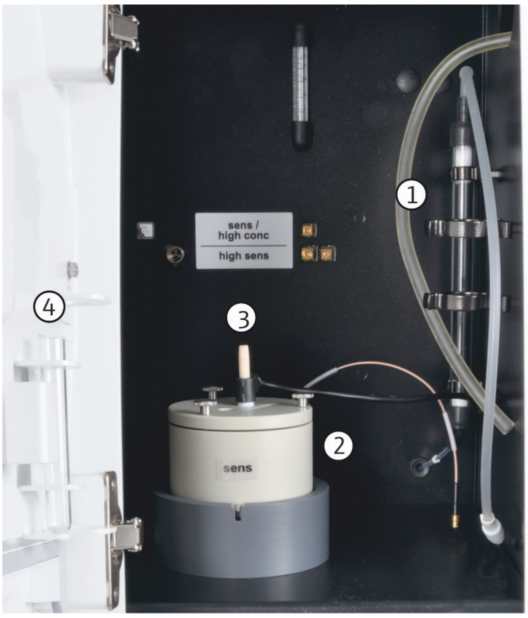


6. Connect the measuring gas hose (4) via the spherical joint adapter to the sulfuric acid container. Secure the connection with a forked clamp.
7. If not already done, route the measuring gas hose through the holes at the top of the basic device's and the chlorine module's side panels.

8. Put the top cover into place and close the doors.

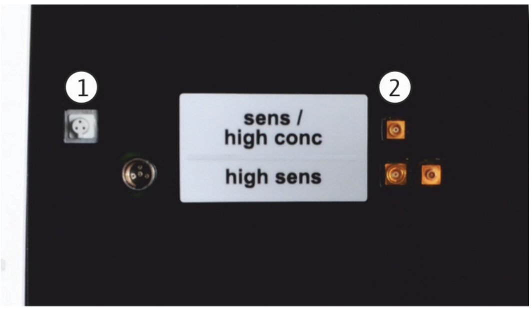
   ✓ The sulfuric acid container (vertical) is mounted.

Preparing the chlorine module:



1. Fill the adsorption tube (1) with activated carbon and insert it into the chlorine module (→ "Replace the activated carbon in the adsorption tube", p. 91).
2. Insert the measuring cell "sensitive" with lid (2) into the chlorine module.
3. Insert the combined electrode into the opening (3) of the measuring cell.
4. Insert the storage container for the combined electrode (4) into the bracket in the door of the chlorine module.

5. Connect the gas outlet of the measuring cell (5) via the olive-tip suction adapter and hose no. 5 to the upper end of the adsorption tube.
6. Connect the lower end of the adsorption tube to the intake pump using hose no. 4.
7. Attach the measuring gas hose to the connection of the combined electrode (6).



8. Electrically connect the combined electrode and the measuring cell to the inside of the rear panel of the chlorine module.
   - Connection combined electrode (1)
   - Connection measuring cell (2)

6.2.2 Preparing the measuring cell

The preparation of the measuring cell includes the following steps:

- Preparation of the electrolyte solution
- Execution of the end point routine

The standard version of the analyzer is equipped with the measuring cell "sensitive". It has the same function as the optional measuring cell "high concentration" which works with larger volumes of electrolyte solution.

Preparation of the electrolyte solution

WARNING

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

Wear the appropriate protective clothing when preparing the electrolyte solution! Observe all instructions and specifications in the safety data sheets!

Reagents required:

- 200 ml of acetic acid c = 100 % (glacial acetic acid)
- 4 ml of concentrated nitric acid
- 4 g of gelatin
- 1.0 g of thymol
- 0.3 g of thymol blue
- 500 ml of methanol

Preparation of the electrolyte solution

The electrolyte solution for the measuring cells "sensitive" and "high concentration" is prepared as follows:

1. 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 up to the marking.

2. 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.

3. Solution B2:

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

4. 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 up to the marking.

5. Solution C – finished electrolyte:

   Pipette 8 ml of solution B into a 100-ml measuring cylinder and top up to 100 ml with solution A, or

   Pipette 40 ml of solution B into a 100-ml measuring cylinder and top up to 500 ml with solution A.

Storage and durability 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.

Execution of 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 coulometric measuring cell is in the range of 1000 to 5000 counts.

1. Start the end point routine via the menu item SYSTEM ▸ END POINT ROUTINE
2. Use hose number 5 to pull the olive-tip suction adapter off the cover of the measuring cell. Upon the prompt meter the HCl solution directly into the measuring cell:
   - Measuring cell "sensitive": 200 μl of a 0.01 N HCl solution
   - Measuring cell "high concentration": 200 μl of a 0.1 N HCl solution
3. Start the end point routine immediately after metering by clicking [OK].
4. The status END POINT ROUTINE is displayed during the process in the window STATUS ANALYZER. After the end point routine the status STAND-BY TITRATION and the current indicator value are displayed in the window STATUS ANALYZER.

   ✓ The system is ready for measurements.

The operating point determined for the combined electrode is displayed under the menu item SYSTEM ▸ COMPONENT TEST in the CL-AMP tab.

Protecting the combined electrode

Observe the following instructions 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.

6.2.3 Performing measures using manual sample feed

WARNING

Concentrated sulfuric acid can cause severe chemical burns! Wear appropriate protective clothing when working at the sulfuric acid container. Observe all specifications in the safety data sheet!

1. Fill the sulfuric acid container (vertical) with fresh sulfuric acid every day. If necessary, remove any used quartz containers from the open combustion tube and clean it.
2. Preparing the measuring cell:
   - Fill the measuring cell with fresh electrolyte solution every day.
     Measuring cell "sensitive": 20 ml
   - Insert the magnetic stirring rod into the measuring cell. Attach the cover and tighten it using the three knurled head screw.
   - Insert the combined electrode into the designated opening and connect it electrically. Electrically connect the measuring cell.
   - Connect the gas transfer hose from the sulfuric acid container in the basic device to the combined electrode. Connect the hose for the adsorption tube (hose no. 5) via the olive-tip suction adapter to the measuring cell "sensitive".
3. The analyzer and the chlorine module are switched on.
   - ✓ The devices are booted. The status LEDs on the front panel lights up in green after approximately 30 seconds.
4. Open the oxygen supply and set the primary pressure to 4 to 6 bar.
5. Switch on the PC.
6. Start the control and analysis software multiWin and log in with user name and password.
7. Initialize the analysis system by clicking on [INITIALIZE ANALYZER].
   - ✓ The initialization and automatic detection of all connected components will be executed.

   In the window DEVICE STATUS any components not yet ready for operation will be shown in red. The time it takes to preheat the furnace to 950 °C is about 30 to 40 min. During the warm-up phase it is not possible to start a measurement.

   If the analyzer is still not ready for measurements after about 40 min., carry out a fault analysis in accordance with the notes in chapter "Fault removal" on page 107.
8. Carry out the end point routine for the measuring cell.
9. If necessary, test the measuring cell using a direct method.
10. Prepare the samples for the manual sample introduction.

    Depending on the sample preparation, these samples may be quartz containers with loaded activated carbon (column method) or frit containers with filter and wet filter cake (batch method).
11. Create a new method under METHOD ▸ METHOD NEW and release it.
12. Activate the new method or an already existing method using the menu command METHOD ▸ METHOD – ACTIVATE.
13. Starting the measurement:
    - Select START ▸ START-ANALYSIS.
    - Select an analysis group or create a new one.

- Create an analysis sequence.
  Enter the sample ID for all samples in the field NAME.
  Confirm the entry with [OK].
- Click the [START MEASUREMENT] button.

✓ The prepared analysis sequence is processed.

14. Follow the prompts in the control and analysis software multiWin and insert the one sample after the other through the oxygen sluice into the combustion tube. Confirm each inserted sample with [OK].

    ✓ The loaded activated carbon is combusted in the oxygen stream and turned into hydrogen halide, carbon dioxide and water. After drying the pyrolysis gas the halide is determined in micro-coulometrically in the chlorine module.

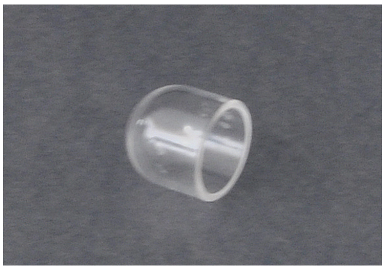
6.2.4 Carrying out measurements with the autosampler autoX 36 / autoX 36d

WARNING

Concentrated sulfuric acid can cause severe chemical burns! Wear appropriate protective clothing when working at the sulfuric acid container. Observe all specifications in the safety data sheet!

1. Connect the autosampler to the "sampler" connection on the rear panel of the multi X 2500 using the interface cable.
2. Fill the sulfuric acid container (vertical) with fresh sulfuric acid every day.

   If necessary, remove any used quartz containers from the open combustion tube.
3. When using the autosampler autoX 36d (with ejection function):



Use tweezers to place the quartz crucible into the combustion tube.

The quartz crucible prevents the quartz wool to form lumps when it directly touches the hot activated carbon. This would prevent it from filtering ashes and soot from the measuring gas stream.

4. Remove the top cover of the analyzer and insert the autosampler without rack into the opening.
5. Preparing the measuring cell:
   - Fill the measuring cell with fresh electrolyte solution every day.
     Measuring cell "sensitive": 20 ml
   - Insert the magnetic stirring rod into the measuring cell. Attach the cover and tighten it using the three knurled head screw.
   - Insert the combined electrode and connect it electrically. Electrically connect the measuring cell.

- Connect the gas transfer hose from the sulfuric acid container in the basic device to the combined electrode. Connect the hose for the adsorption tube (hose no. 5) via the olive-tip suction adapter to the measuring cell "sensitive".

6. The analyzer and the chlorine module are switched on.
   - ✓ The devices are booted. The status LEDs on the front panel lights up in green after approximately 30 seconds.
7. Open the oxygen supply and set the primary pressure to 4 to 6 bar.
8. Switch on the PC.
9. Start the control and analysis software multiWin and log in with user name and password.
10. Initialize the analysis system by clicking on [Initialize analyzer].
    - ✓ The initialization and automatic detection of all connected components will be executed.

    In the window Device status any components not yet ready for operation will be shown in red. The time it takes to preheat the furnace to 950 °C is about 30 to 40 min. During the warm-up phase it is not possible to start a measurement.

    If the analyzer is still not ready for measurements after about 40 min., carry out a fault analysis in accordance with the notes in chapter "Fault removal" on page 107.
11. Carry out the end point routine for the measuring cell.
12. If necessary, test the measuring cell using a direct method.
13. When prompted by the control and analysis software multiWin place the loaded sample rack into the autosampler.

    Position 0 must point toward the combustion tube or the ejector. Carefully rotate the rack until it locks into place. Place the cover on the rack.
14. Create a new method under Method ▶Method new and release it.
15. Activate the new method or an already existing method using the menu command Method ▶ Method – activate.
16. Starting the measurement:
    - Select Start ▶ Start-Analysis.
    - Select an analysis group or create a new one.
    - Create an analysis sequence.
      Enter the sample ID for all samples in the field Name.
      Confirm the entry with [OK].
    - Click the [Start measurement] button.
    - ✓ The prepared analysis sequence is processed. The samples are combusted in the oxygen stream. The halide is determined in the chlorine module.

6.2.5 Installing the pre-combustion adapter

CAUTION

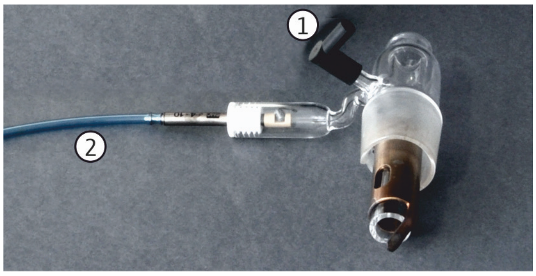
Risk of burns! Only install the combustion tube in the cold state!

NOTICE

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

Do not touch the combustion tube with your hands. Wear protective gloves. If necessary, clean the combustion tube externally before installing it in the combustion furnace (e.g. by wiping it with moist cellulose).

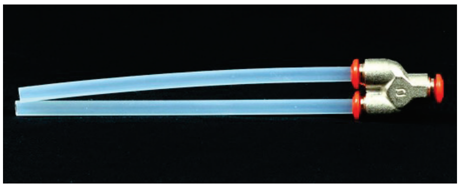
1. Exit the multiWin control software. Allow the multi X 2500 to cool down and switch the device off. Cut the gas supply.
2. Remove the top cover and open the front doors.



3. Replace the oxygen sluice on the open combustion tube by the pre-combustion adapter.
4. Connect the angled adapter (1) with the oxygen hose (hose no. 3) to the adapter.
5. Connect the hose of the pneumatic unit (2) to hose no. 20 in the multi X 2500.

   (Hose no. 20 is attached to a bracket at the top of the housing of the multi X 2500.)
6. Reinsert the top cover. Close the doors.

7. Connect the interface cable of the pneumatic box (3) to the connection "external" (3) on the rear panel of the basic device.
8. Use the gas split (see below) to connect the oxygen connection on the pneumatic box (4) and the basic device (connection IN O_2, 4) to the gas supply.



9. Use the connection hose to link the connection on the pneumatic box (5) to the "Sewage sludge module" connection on the basic device (5).

   ✓ The pre-combustion adapter is installed and can now be controlled using the multiWin software.

6.3 AOX determination in horizontal operating mode

Sample preparation	Column and batch method
Operating mode:	horizontal
Measuring cell	"sensitive"
Measuring range	1 to 100 µg Cl
Electrolyte volume	20 ml
Combustion tube	Multi-purpose combustion tube
manual sample introduction	With manual boat feeder (MBD)
automatic sample introduction	With automatic boat feeder (ABD) for large sample series combined with autosampler autoX 112 with AOX/solids rack (column and batch method)

The multi-purpose combustion tube is used for AOX determination in horizontal operating mode. The transformed hydrogen halides are quantified in the measuring cell "sensitive".

Depending on the sample preparation, these samples may be quartz containers with loaded activated carbon or filters with wet filter cake. The samples are inserted into a quartz glass boats. The quartz glass boat is then introduced into the combustion tube using the manual boat feeder (MBD) or the automatic boat feeder (ABD).

For large sample series it is possible to combine the ABD with the autosampler autoX 112. Installation and commissioning of the ABD and the autoX 112 are described in the respective devices' user manuals.

6.3.1 Preparing the analyzer (optional variant)

The preparation of the analyzer includes the following steps:

- Installing the auto-protection assembly and the gas transfer line
- Inserting the multi-purpose combustion tube
- Prepare the chlorine module

Installing the auto-protection assembly

CAUTION

There is a risk of burns on the combustion furnace! Only install the auto-protection assembly when the device is cold or allow the device to cool down sufficiently!

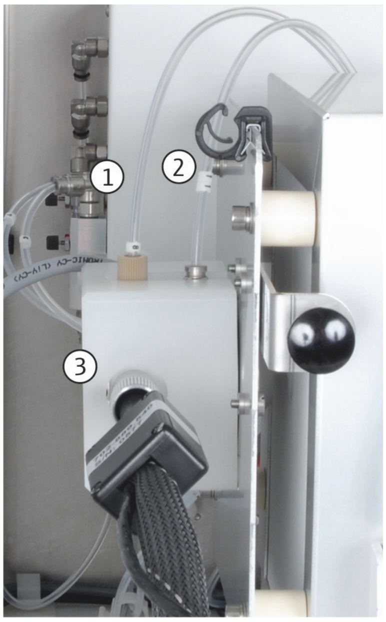
NOTICE

Always switch off all components of the multi X 2500 before electrically connecting them! Connecting or disconnecting electrical contacts may damage the sensitive electronic components of the multi X 2500.

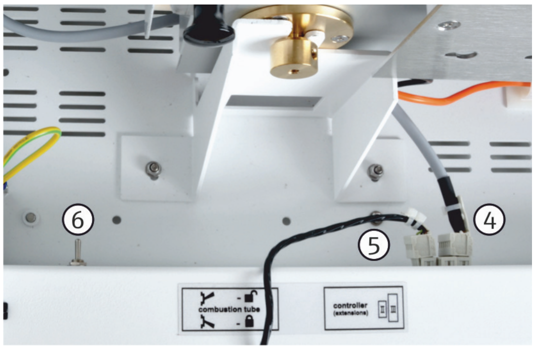
1. Exit the multiWin software. Allow the multi X 2500 to cool down. Then, use the mains switch to switch off the basic device and the chlorine module and cut the gas supply.
2. Tilt the combustion furnace into horizontal position.

3. Hold the auto-protection assembly with the left hand and pull the knob of the clamping mount with the right hand to open the lock.

   Attach the auto-protection assembly to the combustion furnace.



4. Screw hose no. 8 (1) into the connection.
5. Plug in hose no. 11 (2). Push down the ring of the plug-in connector to do this.
6. Screw the gas transfer line (3) into the connection at the side.



7. Insert the electrical connections for the auto-protection assembly (4) and for the gas transfer line (5) into the terminals.
8. Route the gas transfer line through the opening at the top left of the basic device into the chlorine module.
9. Open the pneumatic seal on the auto-protection assembly. Move the toggle switch (6) up so that the combustion tube can be inserted.

Inserting the multi-purpose combustion tube:

WARNING

Danger of explosion! The hose connections for argon and oxygen on the combustion tube must not be mixed up.

CAUTION

Risk of burns! Only install the combustion tube in the cold state!

NOTICE

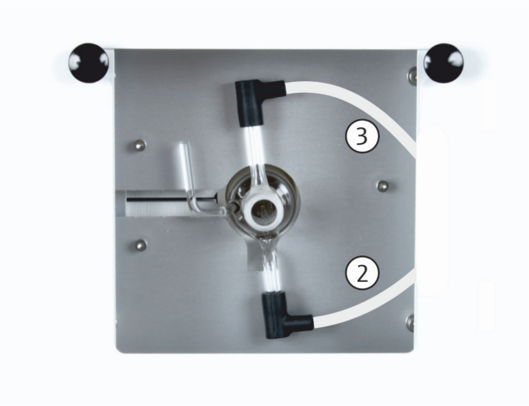
Alkaline salts (hand perspiration) cause crystallizations in the quartz glass when heating the combustion furnace which reduce the service life of the combustion tube.

Where possible do not touch the combustions tube with your hands during installation. Wear protective gloves. If necessary, clean the combustion tube externally before installing it in the combustion furnace (e.g. by wiping it with moist cellulose).

NOTICE

Closing the pneumatic seal when the combustion tube has not been inserted into the furnace may destroy the auto-protection assembly. Only move the toggle switch down if a combustion tube has been inserted.

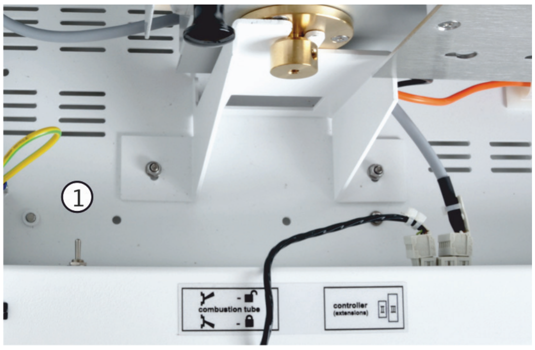
1. Open the service flaps on the right-hand side of the basic device.
2. Insert the multi-purpose combustion tube into the combustion furnace.



3. Connect hose no. 3 (2) and hose no. 4 (3) to the combustion tube. To do this, insert the hose ends into the angled adapters on the combustion tube.

   NOTICE! Do not push the hoses and the connectors beyond the branch length of the angled adapter. Only then, the gas will be able to flow freely.

4. Connect a sample introduction module (MBD, ABD).



5. Switch on the multi X 2500. Open the gas supply.
6. Close the pneumatic seal to the auto-protection assembly. To do this, move the toggle switch (1) down.
   - ✓ This completes the installation of the combustion tube and the locking of the auto-protection assembly.

Preparing the chlorine module:

WARNING

Concentrated sulfuric acid is used as desiccant. The concentrated acid can cause severe chemical burns! Wear protective clothing when working at the sulfuric acid container. Observe the specifications in the safety data sheet!

NOTICE

Risk of damaging the intake pump of the chlorine module!

Make sure to properly connect the gas supply in the measuring cell when using the multi-purpose combustion tube. Never connect the measuring cell to the intake pump via the olive-tip suction adapter and the hose. Otherwise, electrolyte will be drawn into the pump unit.

The hose of the transfer line is connected to the connector at the terminal of the sulfuric acid container using a banjo bolt with conical nipple. Make sure that the conical nipples are seated correctly!

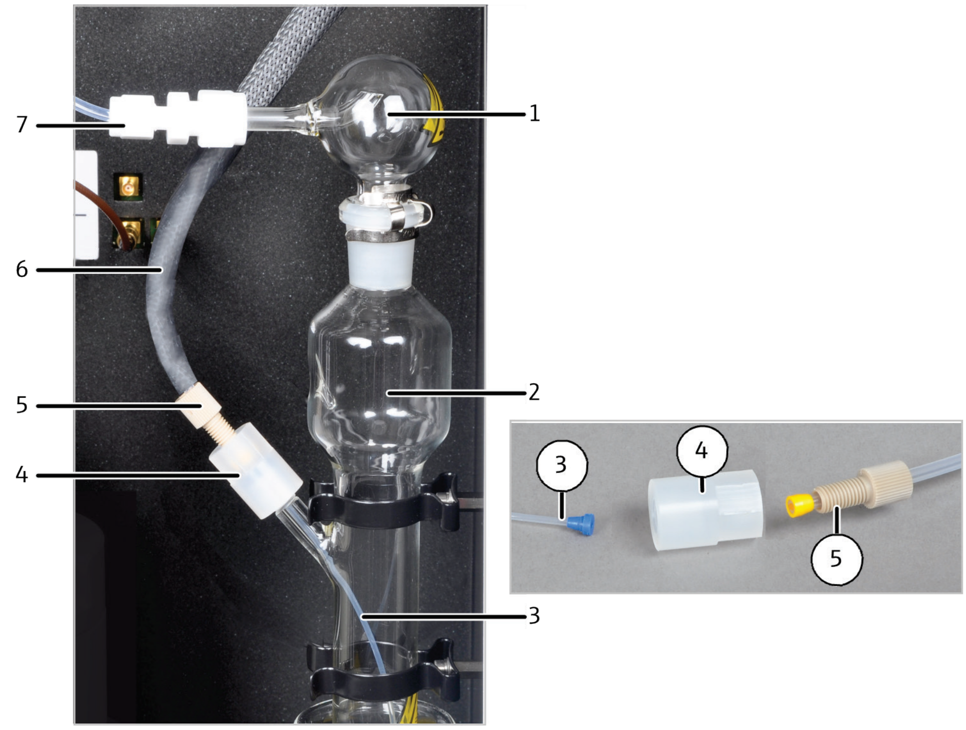
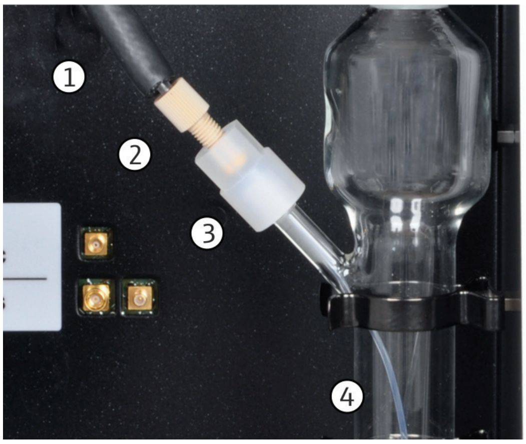


Fig. 45 **Connection transfer line – sulfuric acid container**

1 Safety attachment
2 Sulfuric acid container
3 Hose to introduce measuring gas
4 Connector
5 Banjo bolt
6 Measuring gas transfer to the measuring cell
7 Gas transfer line to measuring cell

1. Place the sulfuric acid container (2 in Fig. 36) somewhere safe and fill it with 20 ml of concentrated sulfuric acid. A large beaker is suitable for safe storage and transport (e.g., 500 ml).
2. Attach a funnel to the sulfuric acid container. Fill 20 ml of concentrated sulfuric acid into the sulfuric acid container.
3. Fit the safety attachment (1) to the sulfuric acid container and attach the safety clip. Connect the gas transfer line to the measuring cell to the safety attachment using the Teflon connector (7).



4. Carefully insert the sulfuric acid container into the bracket.
5. Insert the thin hose (4) into the sulfuric acid container. Make sure that the conical nipple tightly seals the sulfuric acid container.
6. Screw the connector (3) onto the sulfuric acid container.
7. Pass the transfer line (1) through the banjo bolt (2) to connect it with the connector. Make sure that the conical nipple tightly seals the banjo bolt.
8. Place the Petri dish and the beaker under the sulfuric acid container.

Inserting the measuring cell:

NOTICE

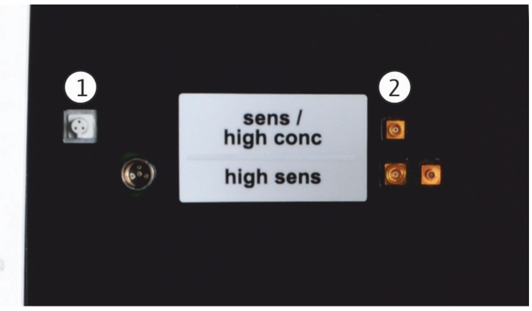
Risk of damaging the intake pump of the chlorine module!

Make sure to properly connect the gas supply in the measuring cell when using the multi-purpose combustion tube. Never connect the measuring cell to the intake pump via the olive-tip suction adapter and the hose. Otherwise, electrolyte will be drawn into the pump unit.

The measuring cells "sensitive" and "high concentration" are inserted to the chlorine module as follows:

1. Insert the measuring cell "sensitive" with lid into the chlorine module. If used, insert the measuring cell "high concentration" into the chlorine module (TX determination).

2. Insert the storage container for the combined electrode into the bracket in the door of the chlorine module.
3. Insert the combined electrode into the opening of the measuring cell.
4. Insert the measuring gas hose from the sulfuric acid container using the Teflon connector (1) into the connection of the combined electrode.
5. Connect the olive-tip suction adapter to the exhaust air hose in the chlorine module.



6. Connecting the combined electrode and the measuring cell at the inside of the rear panel of the chlorine module:
   - Connection combined electrode (1)
   - Connection measuring cell (2)

6.3.2 Preparing the measuring cell

The preparation of the measuring cell for daily measurements includes the following steps:

- Preparation of the electrolyte solution
- Execution of the end point routine

See chapter "Preparing the measuring cell", p. 59 for information on how to prepare the measuring cells "sensitive" and "high concentration".

6.3.3 Performing measurements

WARNING

Concentrated sulfuric acid can cause severe chemical burns! Wear appropriate protective clothing when working at the sulfuric acid container. Observe all specifications in the safety data sheet!

1. Fill fresh sulfuric acid into the sulfuric acid container on the chlorine module every day (→ "Changing the sulfuric acid and cleaning the sulfuric acid container", p. 101).
2. Preparing the measuring cell:
    - Fill the measuring cell with fresh electrolyte solution every day.
      Measuring cell "sensitive": 20 ml
    - Insert the magnetic stirring rod into the measuring cell. Attach the cover and tighten it using the three knurled head screw.
    - Insert the combined electrode and connect it electrically. Electrically connect the measuring cell.
    - Connect the combined electrode to the hose of the sulfuric acid container.
    - Connect the olive-tip suction adapter to the exhaust air hose.

   NOTICE! Never connect the measuring cell to the intake pump via the olive-tip suction adapter and the hose. Otherwise, electrolyte will be drawn into the pump unit.
3. The analyzer and the chlorine module are switched on.
    - ✓ The devices are booted. The status LEDs on the front panel lights up in green after approximately 30 seconds.
4. Open the gas supply and set the primary pressure to 4 to 6 bar.
5. Switch on the PC.
6. Start the control and analysis software multiWin and log in with user name and password.
7. Initialize the analysis system by clicking on [INITIALIZE ANALYZER].
    - ✓ The initialization and automatic detection of all connected components will be executed.

In the window DEVICE STATUS any components not yet ready for operation will be shown in red. The time it takes to preheat the furnace to 950 °C is about 30 to 40 min. During the warm-up phase it is not possible to start a measurement.

If the analyzer is still not ready for measurements after about 40 min., carry out a fault analysis in accordance with the notes in chapter "Fault removal" on page 107.

8. Carry out an end point routine.
9. If necessary, test the measuring cell using a direct method.
10. Providing the samples:

    Load the AOX/solids rack of the autosampler autoX 112 with samples.

    Alternatively, keep the samples at hand for introducing them with via ABD or MBD.
11. Create a new method under METHOD ▸METHOD NEW and release it.
12. Activate the new method or an already existing method using the menu command METHOD ▸ METHOD – ACTIVATE.
13. Starting the measurement:
    - Select START ▸ START-ANALYSIS.
    - Select an analysis group or create a new one.
    - Create an analysis sequence.
      Enter the sample ID for all samples in the field NAME.
      Confirm the entry with [OK].
    - Click the [START MEASUREMENT] button.

    ✓ The prepared analysis sequence is processed.

For manual sample introduction, follow the prompts in the control and analysis software multiWin and introduce the samples one after the other.

7 EOX determination

Sample preparation	Extraction of organically bound halogens using organic solvents	
Operating mode:	vertical and horizontal	
measuring cells	"sensitive"	"high sensitive"
Measuring range	1 to 100 µg Cl	0.01 to 10 µg Cl
Electrolyte volume	20 ml	65 ml
Combustion tube	Multi-purpose combustion tube	
manual sample introduction	via injection port with septum, option: with auto-injector	
automatic sample introduction	vertical: with autosampler autoX 112 (with EOX/liquids rack and dosing syringe) horizontal: with automatic boat feeder (ABD) in combination with autosampler autoX 112 (with EOX/liquids rack and dosing syringe)	

For EOX determination, the samples are prepared in accordance with the extraction methods specified in the standards. The extracts (max. 100 µl) are combusted in the argon/oxygen stream. During this process, they are transformed into hydrogen halides whose mass concentration is determined as chloride in the chlorine module.

The EOX determination can be carried out in both vertical and horizontal operating mode. The multi-purpose combustion tube is employed in both cases.

The transformed hydrogen halides are determined in the measuring cell "high sensitive" or "sensitive" depending on the concentration of the sample. The use of the measuring cell "high sensitive" is recommended for very low EOX contents.

For the EOX determination the extracts are introduced directly to the combustion tube via the injection port. For this purpose, a screw cap with septum is attached to the combustion tube. The auto-injector can be used for exact dosing of individual samples. The autosampler autoX 112 is available for large sample series.

When determining EOX in horizontal operating mode, the autosampler autoX 112 is combined with the automatic boat feeder (ABD). The extracts are injected to quartz glass boats via the injection port with septum at the sluice of the ABD. The ABD then transfers the loaded quartz glass boats to the combustion tube of the multi X 2500.

The samples have a high content of organic solvents. Therefore it is recommended to equip the analyzer with a flame sensor when working in horizontal operating mode. The flame sensor is connected to the ABD and controls the speed with which the boats are introduced. This helps to prevent strong soot formation inside the combustion tube.

Installation and commissioning of the ABD and the autoX 112 are described in the respective devices' user manuals.

7.1 Preparing the analyzer (optional variant)

The preparation of the analyzer includes the following steps:

- Installing the auto-protection assembly and the gas transfer line
- Inserting the multi-purpose combustion tube
- Prepare the chlorine module

Refer to chapter "AOX determination in horizontal operating mode", p. 65 for information on the installation of the auto-protection assembly and the gas transfer line.

Inserting the multi-purpose combustion tube:

The following section describes how the multi-purpose combustion tube is inserted in horizontal and vertical operating mode.

WARNING

Danger of explosion! The hose connections for argon and oxygen on the combustion tube must not be mixed up.

CAUTION

Risk of burns! Only install the combustion tube in the cold state!

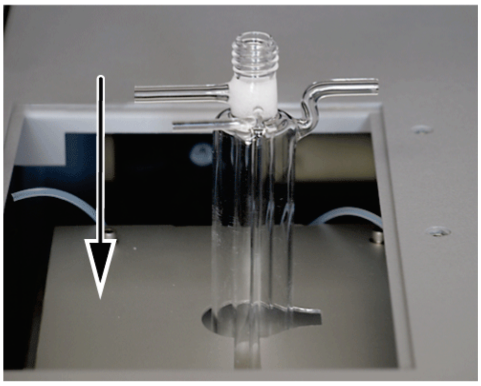
Note

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

Where possible do not touch the combustions tube with your hands during installation. Wear protective gloves. If necessary, clean the combustion tube externally before installing it in the combustion furnace (e.g. by wiping it with moist cellulose).

1. Make sure that the analyzer is switched off. Switch off the gas supply.
2. In vertical operating mode, remove the cover at the top. In horizontal operating mode, open the service flap on the right-hand side.
3. Insert the combustion tube into the combustion furnace. When doing this, make sure that the connections on the combustion tube fit into the recesses on the furnace.

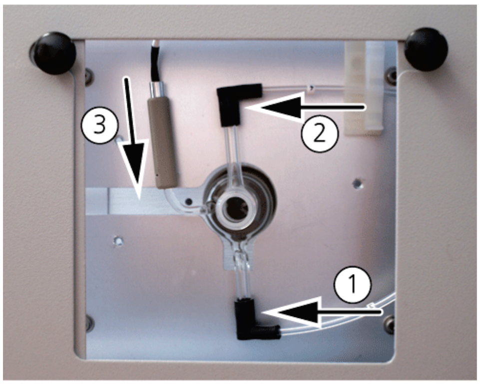
Horizontal operating mode



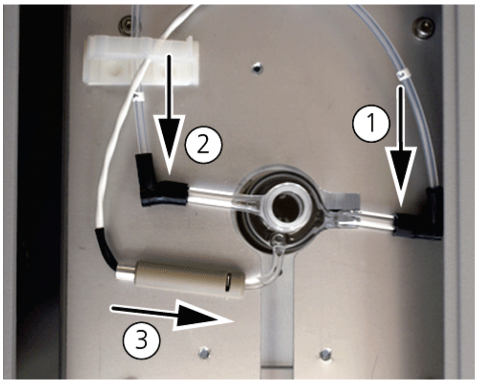
Vertical operating mode

4. Open the gas supply.
5. Connect hose no. 3 (1) and hose no. 4 (2) to the combustion tube. To do this, insert the hose ends into the angled adapters on the combustion tube.

   ATTENTION! Do not push the hoses and the connectors beyond the branch length of the angled adapter. Only then, the gas will be able to flow freely.



Horizontal operating mode



Vertical operating mode

6. Plug in the flame sensor (3) to the connection, if used.
7. Screw a screw cap with septum to the combustion tube.

8. Close the pneumatic seal to the auto-protection assembly. To do this, move the toggle switch (1) down.
   - ✓ The combustion tube is now locked in the auto-protection assembly.
9. In vertical operating mode, put the cover at the top back into place.
10. Connect a sample introduction module.

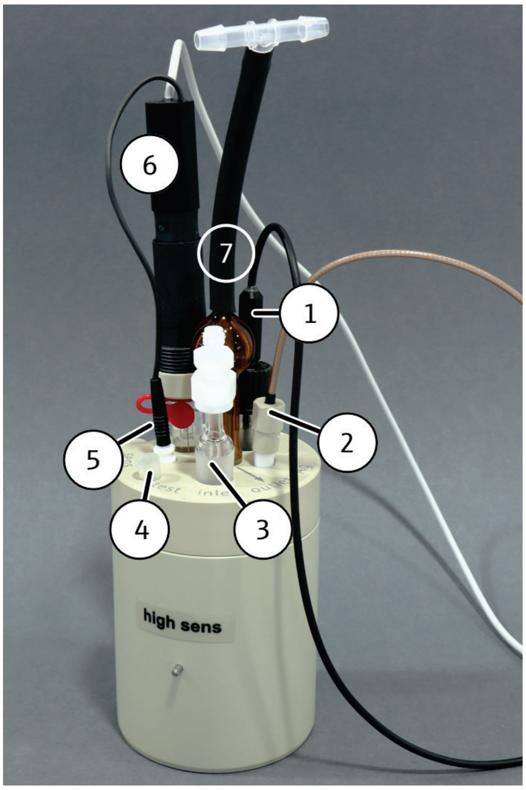
Prepare the chlorine module

For the EOX determination, the sulfuric acid container is inserted into the chlorine module. Refer to chapter "AOX determination in horizontal operating mode", p. 65 for information on the installation of the sulfuric acid container.

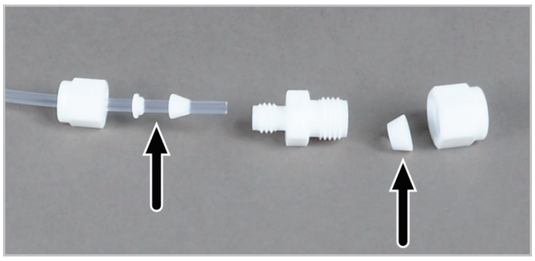
It is recommended to use the measuring cell "high sensitive" for analyzing samples with low EOX content. The installation of this measuring cell is described in the following section. The use of the measuring cell "sensitive" is described under AOX determination in horizontal operating mode.

The measuring cells "high sensitive" is inserted to the chlorine module as follows:

1. Insert the measuring cell "high sensitive" with magnetic stirring rod and lid into the chlorine module.
2. Fill the measuring cell with electrolyte (→ "Preparing the measuring cell", p. 77).

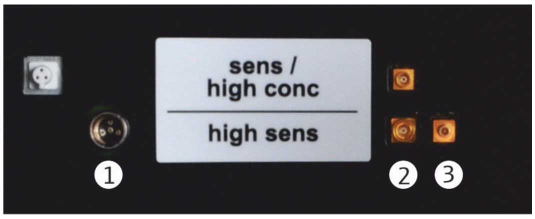


3. Insert the components in the following openings:
   Platinum electrode with salt bridge (1): "Pt" opening
   Silver electrode (2): "Ag" opening
   Gas inlet hose (3): "Inlet" opening
   Sensor electrode (5): "sens" opening
   reference electrode (6): large "ref" opening
   adapter (7): medium "outlet" opening
   The openings on the lid align the sensor electrode and the reference electrode with each other.
4. Close the opening for direct injection (4, "test") with a plug.
5. Connect the short hose with the T piece to the adapter (7). Connect the exhaust hose in the detection module with a limb of the T piece.
6. Place the pre-amplifier on the reference electrode. Connect the pre-amplifier with the sensor electrode.



7. Connect the measuring gas hose from the sulfuric acid container to the gas inlet tube (4) using the Teflon screw connection.

   ATTENTION! The two conical nipples of the Teflon screw connection must fit tightly to the hose. Otherwise, the gas-tightness of the connection cannot be guaranteed.



8. Connecting the electrodes at the inside of the rear panel of the chlorine module:
   - Connection sensor electrode (1)
   - Connection silver electrode (2)
   - Connection platinum electrode (3)

7.2 Preparing the measuring cell

The preparation of the measuring cell for daily measurements includes the following steps:

- Preparing the electrolyte solution
- Performing the end point routine

This section describes the preparation of the measuring cell "high sensitive". See chapter "Preparing the measuring cell", p. 59 for information on how to prepare the measuring cells "sensitive" and "high concentration".

Preparing the electrolyte solution

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

WARNING

100 % acetic acid (glacial acetic acid) can cause severe chemical burns! Wear the appropriate protective clothing when preparing the electrolyte solution! Observe all instructions and specifications in the safety data sheet!

Reagents required:

- 800 ml of acetic acid c = 100 % (glacial acetic acid)
- 2.7 g of sodium acetate p.a., anhydrous

Preparing the electrolyte solution

Proceed as follows to prepare 1 l of electrolyte solution for the measuring cell "high sensitive":

1. Dilute 2.7 g of sodium acetate (CH_3COONa) in 200 ml of ultrapure water in a 1 liter volumetric flask.
2. Carefully add 800 ml of glacial acetic acid. Keep moving the flask while pouring the acid. Carefully shake the mixture.

   ATTENTION! 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:

- lower limit: 1000 counts
- upper limit: 10000 counts
- optimum operating point: 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 DEVICE STATUS window during this routine.

- Indicator value greater than 10000:

Silver ions are automatically produced. The value set for the operating point of the electrolyte is 3000.

- Indicator value less than 1000:

The software prompts the user to add a dose of 100 µl HCl (10 mg/l) to the measuring cell. If the indicator value rises above 10000, silver ions will be produced automatically and the electrolyte will be set to an operating point of 3000.

After the end point routine, the measuring cell requires approx 15 min to achieve a stable cell potential. During this time, a negative drift with indicator values <3000 counts may be observed.

Manual end point routine

For indicator values within the limits of the operating range (1000 to 10000), the end point routine can be triggered manually. The routine is started in the control and analysis software using the menu command SYSTEM ▸ END POINT ROUTINE.

After the end point routine is completed, the DEVICE STATUS window will show the current indicator value. The system is ready for measurements.

7.3 Performing measurements

WARNING

Concentrated sulfuric acid and concentrated acetic acid can cause severe chemical burns! Wear the appropriate protective clothing when pouring the electrolyte solution! Observe all instructions and specifications in the safety data sheets!

Note

If the gas supply fails while the system is running, e.g., due to an emergency shutdown, or if the gas pressure drops due to a blockage while the system is running or cooling down, sulfuric acid may be blown back into the analysis system. Disconnect the heated transfer line from the sulfuric acid container immediately if the shielding gas supply is not ensured.

1. Fill fresh sulfuric acid into the sulfuric acid container on the chlorine module every day (→ "Changing the sulfuric acid and cleaning the sulfuric acid container", p. 101).
2. Fill the measuring cell with fresh electrolyte solution daily:
   - Discard the used electrolyte solution and clean the measuring cell.
   - Fill the measuring cell with electrolyte solution.
     Measuring cell "high sensitive": approx. 65 ml
   - Insert the magnetic stirring rod into the measuring cell. Fit the cover.
   - Insert the electrodes and connect them electrically.
   - Connect the measuring gas hose from the sulfuric acid container to the gas inlet tube using the Teflon screw connection.
   - Connect the adapter to the extraction hose.

   ATTENTION! Do not connect the measuring cell "high sensitive" to the intake pump. Otherwise, electrolyte could get drawn into the pump.
3. Top up the bridge electrolyte in the reference electrode up to the refill opening every day. Recommendation: Always change the bridge electrolytes when fresh electrolyte solution is prepared.
4. The analyzer and the chlorine module are switched on.
   - ✓ The devices boot up. The status LEDs on the front panel lights up in green after approximately 30 seconds.
5. Open the gas supply and set the primary pressure to 4 to 6 bar.
6. Switch on the PC.
7. Start the control and analysis software multiWin and log in with user name and password.
8. Initialize the analysis system by clicking on [INITIALIZE ANALYZER].
   - ✓ The initialization and automatic detection of all connected components will be executed.

   In the window "Device status" any components not yet ready for operation will be shown in red. The time it takes to preheat the furnace to 950 °C is about 30 to 40 min. During the warm-up phase it is not possible to start a measurement.

   If the analyzer is still not ready for measurements after about 40 min., carry out a fault analysis in accordance with the notes in chapter "Fault removal" on page 107.
9. Carry out an end point routine to adjust the electrolyte to the optimal operating range of the measuring cell.
10. If necessary, test the measuring cell using a direct method.
11. Providing the samples:

    Load the EOX/liquids rack of the autosampler autoX 112 with samples.

    Alternatively, keep the samples at hand for introducing them manually with a syringe or an auto-injector.
12. In the METHOD ▸ NEW METHOD menu, create and release a new method.

13. Activate the new method or an existing method via the menu item METHOD ▸ ACTIVATE METHOD.

14. Start the measurement:

    - Select START ▸ START ANALYSIS.
    - Select an analysis group or create a new one.
    - Create an analysis sequence.
      Enter the sample ID for all samples in the field NAME.
      Confirm the entry with [OK].
    - Click on the [START MEASUREMENT] button.

    ✓ The prepared analysis sequence is processed.

For manual sample introduction, follow the prompts in the control and analysis software multiWin and introduce the samples one after the other.

8 TX/TOX determination

Sample preparation	none		
Operating mode:	horizontal vertical (for liquid, highly vaporizable samples which can be metered with a syringe)		
Measuring cell	"sensitive"	"high sensitive"	"high concentration"
Measuring range	1 to 100 µg Cl	0.01 to 10 µg Cl	10 to 1000 µg Cl
Electrolyte volume	20 ml	65 ml	120 ml
Combustion tube	Multi-purpose combustion tube		
automatic sample introduction	Transferring solid samples with an automatic boat feeder (ADB), for large sample series in combination with autosampler autoX 112 (with AOX/solids rack) Transferring liquid samples with an auto-injector, for large sample series in combination with autosampler autoX 112 (with EOX/liquids rack)		

The analyzer multi X 2500 allows determining the total chlorine content (TX) and/or the total content of organically bound chlorine (TOX) in solid and liquid samples. Solid and highly viscous liquid samples (max. 100 mg) are weighed in in quartz glass boats. Liquid highly vaporizable samples (max. 100 µl) can be metered to the combustion tube using µl-syringes. The samples are combusted in an argon/oxygen stream without any further sample preparation. During this process, organic halogenated compounds are transformed into hydrogen halides. Their mass concentration is determined as chloride.

Depending on the sample matrix, the TX determination is carried out in horizontal operating mode or vertical operating mode. In horizontal operating mode the samples are introduced to the multi-purpose combustion tube by means of an automatic sample feeder. In vertical operating mode an auto-injector or the autosampler autoX 112 meters liquid samples directly into the multi-purpose combustion tube. The vertical operating mode is primarily used for analyzing pure fuels and solvents. The transformed hydrogen halides are quantified in one of the three measuring cells. Which of the three cells they are analyzed in depends on their chlorine content.

When analyzing organic samples in horizontal operating mode it is highly recommended to equip the analyzer with the optional flame sensor. The flame sensor is connected to the ABD and controls the speed with which the boats are introduced. This helps to prevent strong soot formation inside the combustion tube.

9 POX determination

Sample preparation	Purging organically bound halogens in the argon stream, argon supply via gas outlet "ABD OUT" on the basic device
Operating mode:	vertical
Measuring cell	"sensitive"
Measuring range	1 to 100 µg Cl
Electrolyte volume	20 ml
Combustion tube	Open combustion tube
Sample supply	via POX sluice as an attachment to the combustion tube Auto-injector and autosampler cannot be used

For the POX determination organically bound halogens contained in the water sample are purged with argon under defined conditions and combusted in the oxygen stream. During this process, they are transformed into hydrogen halides whose mass concentration is determined as chloride.

The analyzer is operated in its default configuration in vertical operating mode with open combustion tube and measuring cell "sensitive". Refer to chapter "AOX determination in vertical operating mode (recommended)" on page 54 for information on the preparation of the analyzer and the measuring cell and the execution of the measurements.

The connection of the sample container to the multi X 2500 is shown in the hose diagram and described in the following section. The temperature of the sample container is controlled in a water bath outside the analyzer. The user is responsible for properly carrying out this process.

9.1 Performing measurements

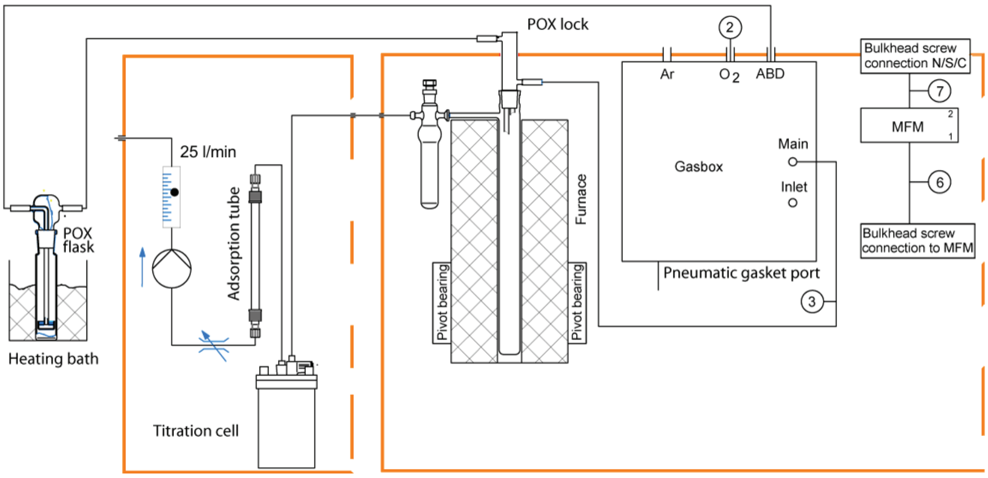


Fig. 46 Hose diagram POX determination

WARNING

Organically bound halogens must only be purged with argon! Otherwise, there is a risk of explosion! The purging equipment must only be connected to the "ABD OUT" terminal on the rear panel of the basic device! Before starting the measurement, verify that the purging equipment is connected to the right terminal!

WARNING

Concentrated sulfuric acid can cause severe chemical burns! Wear appropriate protective clothing when working at the sulfuric acid container. Observe all specifications in the safety data sheet!

CAUTION

Risk of burns when removing the oxygen sluice! The oxygen sluice will get very hot during the operation of the combustion furnace! Wear protective gloves when removing the oxygen sluice!

1. Fill the sulfuric acid container (vertical) with fresh sulfuric acid every day.

   If necessary, remove any used quartz containers from the open combustion tube and clean it.
2. Place the top cover on top of the analyzer.
3. Preparing the measuring cell:
   - Fill the measuring cell with fresh electrolyte solution every day.
     Measuring cell "sensitive": 20 ml
   - Insert the magnetic stirring rod into the measuring cell. Attach the cover and tighten it using the three knurled head screw.
   - Insert the combined electrode and connect it electrically. Electrically connect the measuring cell.
   - Connect the gas transfer hose from the sulfuric acid container in the basic device to the combined electrode. Connect the hose for the adsorption tube (hose no. 5) via the olive-tip suction adapter to the measuring cell "sensitive".
4. The analyzer and the chlorine module are switched on.
   - ✓ The devices are booted. The status LEDs on the front panel lights up in green after approximately 30 seconds.
5. Open the gas supply and set the primary pressure to 4 to 6 bar.
6. Switch on the PC.
7. Start the control and analysis software multiWin and log in with user name and password.
8. Initialize the analysis system by clicking on [INITIALIZE ANALYZER].
   - ✓ The initialization and automatic detection of all connected components will be executed.

In the window DEVICE STATUS any components not yet ready for operation will be shown in red. The time it takes to preheat the furnace to 950 °C is about 30 to 40 min. During the warm-up phase it is not possible to start a measurement.

If the analyzer is still not ready for measurements after about 40 min., carry out a fault analysis in accordance with the notes in chapter "Fault removal" on page 107.

9. Carry out the end point routine for the measuring cell.
10. If necessary, test the measuring cell using a direct method.
11. Switching from the oxygen sluice to the POX sluice:

    CAUTION! Risk of burns when touching the oxygen sluice and the combustion tube. Wear protective gloves!

    - Remove the top cover.
    - Pull the angled adapter off the oxygen sluice. Remove the oxygen sluice.
    - Attach the POX sluice to the top of the combustion tube.
    - Connect the oxygen hose (2, hose no. 3) to the POX sluice.
    - Connect the measuring gas hose (1, hose from the outlet to the POX cylinder) to the POX sluice.

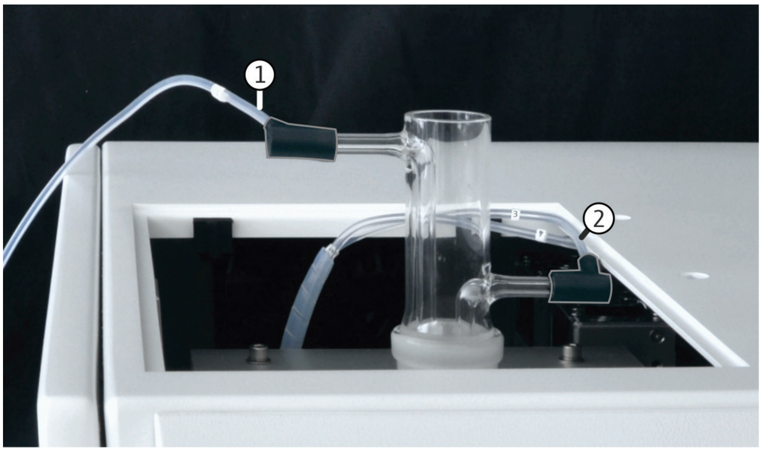


Fig. 47 Connections on the POX sluice

1 Measuring gas hose connection
2 Oxygen hose connection

12. Connecting the argon hose to the POX container:

    WARNING! Observe the following instructions when connecting the POX container – risk of explosion!

    Connect the hose from the inlet of the purging equipment (2) to the "ABD OUT" connection (1) on the rear panel of the basic device.

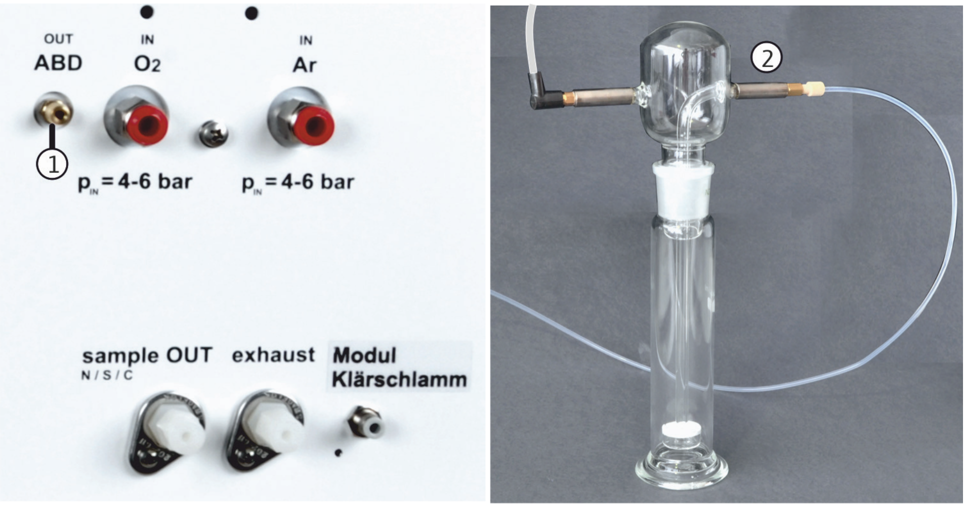


Fig. 48 Connection of the argon hose to the basic device and the POX container

13. Create a new method under METHOD ▸ METHOD NEW and release it.
14. Defining the analysis parameters: sample volume, furnace temperature, POX purging time, dosing time, delay time, integration time.

    The measurement time is the sum of all the configured times.

15. Activate the new method or an already existing method using the menu command METHOD ▸ METHOD – ACTIVATE.
16. Starting the measurement:
    - Select START ▸ START-ANALYSIS.
    - Select an analysis group or create a new one.
    - Create an analysis sequence.
      Enter the sample ID for all samples in the field NAME.
      Confirm the entry with [OK].
    - Click the [START MEASUREMENT] button.
    - ✓ The prepared analysis sequence is processed.

Follow the prompts in the multiWin control and analysis software, attach the purging equipment to the sample container and confirm with [OK].

10 Maintenance and care

10.1 Maintenance intervals

Maintenance item	Task	Maintenance interval
Analyzer	Check gas flow	daily
	Check system tightness	daily
	Cleaning and servicing the device	Weekly
	Check all screw connections for tight fit Replace faulty hoses and connections	Weekly If required (leakage)
	Check fastening screws for tight fit	Monthly
	Replacing the particle filters in the gas inlets	If required (if target values of the gas flows can no longer be set or in case of leaks)
Combustion tube	Check for cracks and damages	Monthly
	Perform a visual inspection of the quartz wool plug at the bottom of the combustion tube and replace if required	After emptying the combustion tube or once per week
	Renew (replace) combustion tube	If required (crystallization, cracks)
	Check angled adapter in the oxygen sluice for tight fit, cracks and damage	Monthly
	Check inspection port with septum for tightness, replace septum (if using the multi-purpose combustion tube)	Daily, as required
Sulfuric acid container	Replace sulfuric acid	As required, at least once per day
	Clean and dry the sulfuric acid container	Weekly For trace analysis with measuring cell "high sensitive" each time the sulfuric acid is changed, otherwise the analytic performance is affected
	Clean and dry the safety attachment	Weekly
	Clean and dry the measuring gas hose	Weekly
Chlorine module	Replace the adsorption agent in the adsorption tube	After 40 hours of operation or once per week
	Check the intake flow	Daily, if required readjust the intake flow to 20 l/h In case of a significant decrease of the intake flow search for obstructions and leaks
	Clean measuring cell	Daily with electrolyte change
	Replace electrolyte	On measurement days or after 8 hours of measurement
	Reference electrode (if using the measuring cell "high sensitive") ■ Check the bridge electrolyte fill level of the reference electrode, refill as necessary ■ Replace bridge electrolyte	 daily Always when fresh electrolyte solution is prepared.

Maintenance item	Task	Maintenance interval
Auto-protection assembly	(if using the multi-purpose combustion tube)	
	Check filters, replace if required	Every three months, replace if required: ■ if filter is sooted, soiled or torn ■ if sulfuric acid was drawn into the APB after an emergency shutdown
	Check pneumatic seal and replace if required	If gas escapes from pneumatic seal (audible hiss)

10.2 Replace particle filters in the gas inlets

WARNING

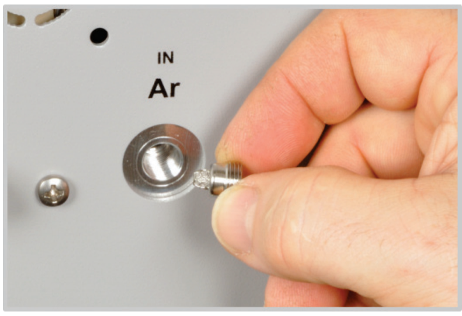
Risk of explosion and fire when swapping the gas connections! Always connect the gas lines correctly and set the required gas pressures.

The gas inlets "Ar" and "O_2" on the rear of the multi X 2500 are equipped with a particle filter. The particle filter must be replaced once the process gases can no longer be set to the target value (check for messages in the software) and potential leaks within the system have been excluded.

1. Switch off the multi X 2500 and the chlorine module and pull the mains plug from the connections.
2. Cut the gas supply at the shut-off valve.
3. Disconnect the chlorine module and the sample introduction module from the multi X 2500 and carefully push them to the side to gain access to the rear of the device.
4. Pull the gas hose from the connection terminal on the rear panel of the multi X 2500. When doing this, press the red ring.

5. Unscrew the gas connections using a 13-mm open-end wrench.

6. Unscrew the particle filters on the inside using a 5-mm Allen key.



7. Insert and tighten the new particle filter.
8. Screw in the gas connections and tighten them with an open-ended wrench.
9. Connect the gas hoses. Make sure to attach the gas hoses to the correct connection terminal.
10. Reconnect the chlorine module and the sample introduction module.
11. Open the gas supply.
12. Plug in the mains cables to the connections on the multi X 2500 and the chlorine module and switch on the devices using the mains switch.
    - ✓ The multi X 2500 is ready for operation again.

10.3 Maintenance activities (standard version)

10.3.1 Removing and cleaning the open combustion tube

WARNING

Concentrated sulfuric acid can cause severe chemical burns! Wear protective clothing when working at the sulfuric acid container. Observe all specifications in the safety data sheet!

CAUTION

Risk of burns! Only remove the combustion tube when the device is cold!

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

CAUTION

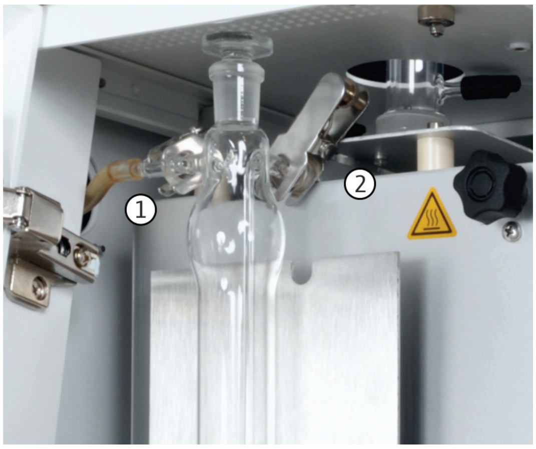
When working with quartz wool avoid the creation of dust! Inhaled dust might cause irritation to respiratory tracts.

NOTICE

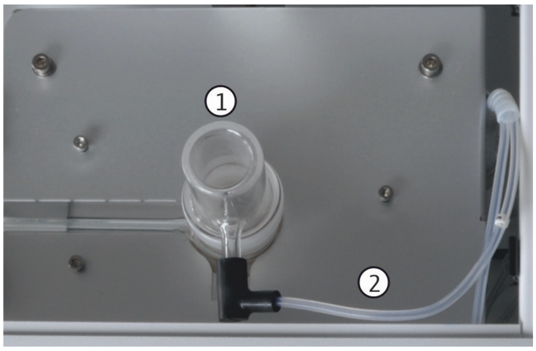
Alkaline salts (hand perspiration) cause crystallizations in the quartz glass when heating the combustion furnace which reduce the service life of the combustion tube.

Do not touch the combustion tube with your bare hands during re-installation. Wear protective gloves. If necessary, clean the combustion tube externally before installing it in the combustion furnace (e.g. by wiping it with moist cellulose).

1. Exit the multiWin control software and allow the analyzer to cool down.
2. Switch off the multi X 2500 and the chlorine module and pull the mains plugs from the power outlet. Cut the gas supply.
3. Remove the top cover or the multi X 2500 or the sample introduction module and open the front doors.



4. Detach the connection hose and the spherical joint (1) from the sulfuric acid container (vertical).
5. Detach the forked clamp (2) between the sulfuric acid container and the combustion tube.
6. Carefully remove the sulfuric acid container from the drip tray and empty it. Observe the regulations for handling concentrated sulfuric acid!



7. Remove the oxygen sluice (1). Pull the angled adapter (2) off the oxygen sluice.
8. Remove the heat bridge from the analyzer.
9. Pull the open combustion tube upwards to remove it from the furnace.

10. Check the combustion tube for excessive crystallization, cracks and blow-outs. Only reuse intact combustion tubes.
11. Remove the quartz wool plug from the combustion tube and check its condition.
12. Thoroughly rinse the empty combustion tube with ultrapure water and dry it well.
13. Carefully push the previously removed quartz wool plug or a new plug into the combustion tube until it reaches the bottom.

The installation of the combustion tube is done in reverse order.

10.3.2 Change the sulfuric acid and clean the sulfuric acid container (vertical)

WARNING

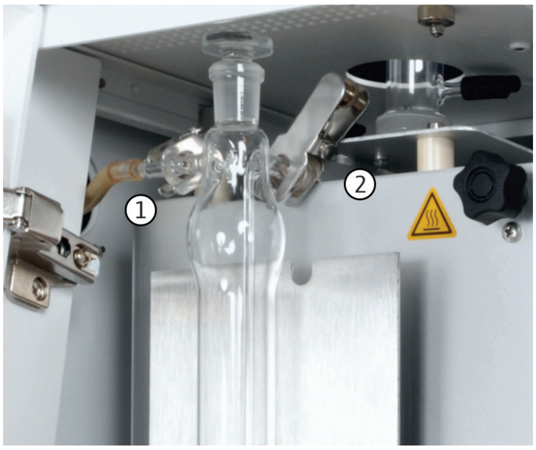
Concentrated sulfuric acid can cause severe chemical burns! Wear appropriate protective clothing when working at the sulfuric acid container. Observe all specifications in the safety data sheet!

CAUTION

Risk of burns! Only remove the sulfuric acid container when the device is cold!

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. As a result, the measured AOX contents will be too low and widely scattered. For this reason, the sulfuric acid must be replaced once per day. High sample throughput may require more frequent replacement.

1. Exit the multiWin control software and allow the analyzer to cool down.
2. Switch off the multi X 2500 and the chlorine module and pull the mains plugs from the power outlet. Cut the gas supply.
3. Remove the top cover or the sample introduction module and open the front doors.



4. Detach the connection hose and spherical joint (1) from the sulfuric acid container (vertical).
5. Detach the forked clamp (2) between the sulfuric acid container and the combustion tube.
6. Carefully remove the sulfuric acid container from the drip tray and empty it. Observe the regulations for handling concentrated sulfuric acid!
7. Rinse the sulfuric acid container with distilled water several times. After that, rinse the container with ethanol. Then dry it (e.g. by blowing it through with an inert gas).
8. Fill the sulfuric acid container with approx. 40 ml of concentrated sulfuric acid. Insert the plug to the container.

The installation of the sulfuric acid container is done in the reverse order.

10.3.3 Replace the activated carbon in the adsorption tube

The activated carbon must be replaced once per week or after approx. 40 operating hours.

1. Exit the multiWin control software and switch off the analyzer.
2. Switch off the basic device and the Cl module from the mains switch. Turn off the gas supply.
3. Pull the adsorption tube out of its mount.
4. Pull the hoses including the plugged in connectors at the top and the bottom off the glass tube.
5. Remove the cotton plug and carefully pour out the adsorption agent.
6. Seal one side of the glass tube with cotton wool and add new activated carbon.
7. Seal the glass tube with cotton wool.
8. Reinsert the two connectors to the filled adsorption tube in the chlorine module.
9. Press the adsorption tube into its mount.

NOTICE! Do not twist or kink the hoses!

10.3.4 Servicing the measuring cell

Since the continuous introduction of hot reaction gases during operation causes electrolyte components to evaporate, it is recommended to change the electrolyte solution once per day:

1. Remove the used electrolyte.
2. Rinse the measuring cell and the magnetic stirring rod with distilled water and with ethanol.
3. Then, carefully wipe out the measuring cell and wipe the magnetic stirring rod with cellulose to remove any silver chloride deposits.
4. Fill the measuring cell with fresh electrolyte:

Measuring cell "sensitive":	20 ml
Measuring cell "high concentration":	approx. 120 ml

Also observe the following notes:

- If the system is decommissioned for several days, clean the measuring cell and store it dry.

- Regularly check the casing of the magnetic stirring rod for cracks. Metal ions entering the electrolyte solution can distort the analysis.
- Avoid fluids from entering the stirring/cooling block and the plug-in contacts (risk of short circuit).

10.3.5 Clean and store the combined electrode

NOTICE

Detergents and abrasives can destroy the combined electrode! The combined electrode consists of ceramic materials which is mechanically sensitive especially near the fused electrodes. For cleaning, the combined electrode may only be rinsed with Ethanol and distilled water.

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 interruptions of operation (one day to the next):
  Store the combined electrode in fresh electrolyte solution.
- For decommissioning over several days:
  Carefully rinse the combined electrode with ethanol and subsequently rinse it with ultrapure water.
  For intensive cleaning, immerse the electrode in Ethanol over-night, stirred with a magnetic stirrer. Do not connect the measuring cell or the electrode to their electrical connections for this.
  Wipe the combined electrode with cellulose and store it in dry condition.

Pulling the combined electrode out of the cover of the measuring cell and connecting it to the measuring gas can result in great mechanical stress if handled incorrectly and lead to its electrical connection becoming fractured.

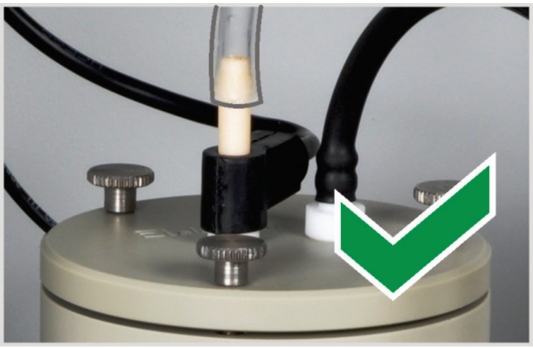
Correct

Hold the electrode from the top and pull the electrode straight upwards out of the cover.

Incorrect

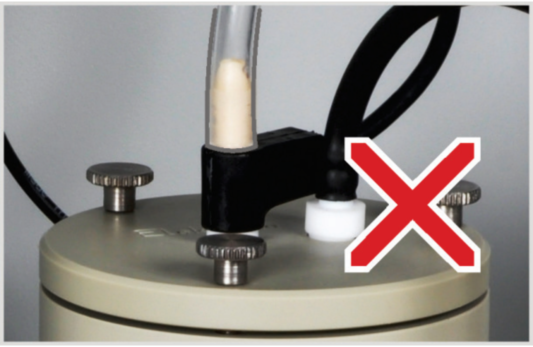
Do not pull and jolt the lateral connection sleeve for the electrical connections. The connections in the sleeve will break (not visible from outside)!

Correct



Only push the measuring gas hose approx. 1 cm onto the electrode. This ensures gas tightness.

Incorrect



If you push the hose too far onto the electrode, it will get stuck on the electrode. When pulling off the hose, the electrode might break!

10.4 Maintenance activities (optional version)

10.4.1 Checking the settings of the gas flows

1. Switch on the system components of the multi X 2500.
2. Open the gas supply to the basic device.
3. Start the multiWin control and analysis software.
4. Enable a method.
   - ✓ The DEVICE STATUS window displays the current gas flows:

EOX determination		
MFC	**Should**	**Description**
MFC 1	300 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 inlet at the combustion tube, value is set in the method
MFC 3	100 ml/min (idle state)	Pyrolysis gas (argon), hose 4 inlet at the combustion tube, value is set in the method

Faulty gas inlet flows are highlighted in red in the DEVICE STATUS window.

10.4.2 Checking the system for tightness

NOTICE

Risk of destroying the internal MFM by corrosive gases! Only use the flow monitoring set included in the scope of delivery to check the system tightness. Make sure that the sulfuric acid container is completely empty.

The system tightness is not tested automatically. The flow monitoring set delivered with the device allows the user to test the system's tightness manually. The test establishes whether the supplied gas flow corresponds to the target flow rate. This allows detecting leaks and obstructions.

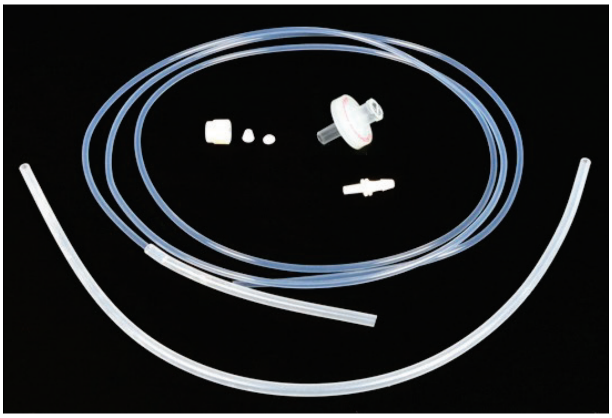


Fig. 49 Flow monitoring set

1. Open the front doors of the analyzer and the chlorine module.
2. Remove the entire sulfuric acid from the sulfuric acid container in the chlorine module. Clean and dry the sulfuric acid container (→ "Changing the sulfuric acid and cleaning the sulfuric acid container", p. 101).

   NOTICE! Always clean and dry the sulfuric acid container. Check the hose system for deposits of acid in form of small drips. If required, clean the hose system. Even minor traces of sulfuric acid in the gas path will damage the MFM.
3. Disconnect the measuring gas hose from the gas inlet tube (measuring cell "high sensitive") or from the combined electrode (measuring cell "sensitive" / "high concentration").
4. Remove the "MFM in" screw connection above the cover of the control electronics in the analyzer.
5. Use the flow monitoring set to link the "MFM in" connection (1) with the measuring gas hose (2) in the chlorine module.

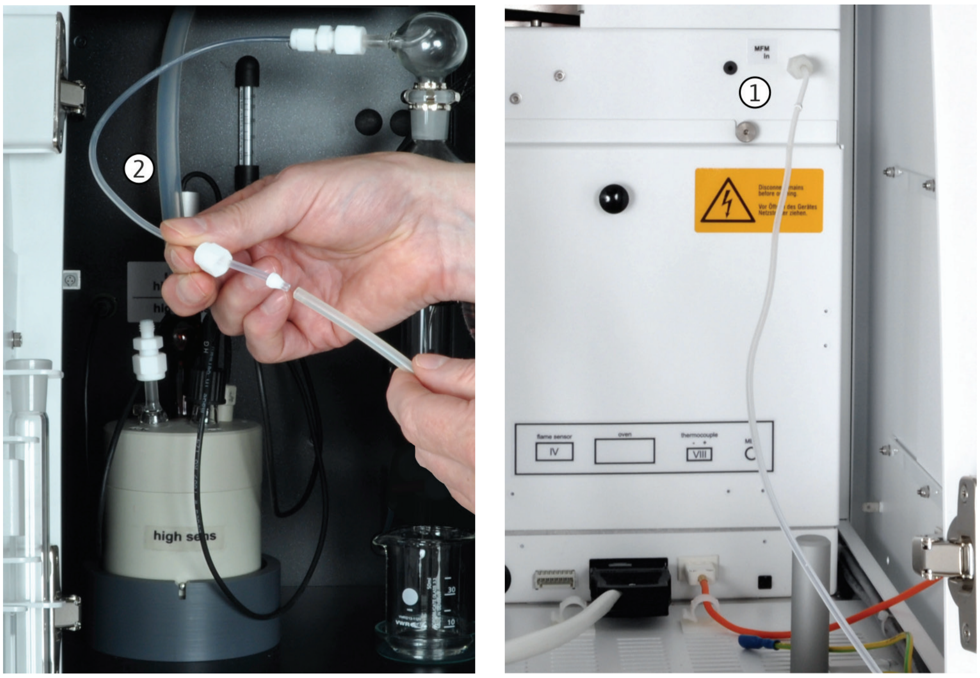


Fig. 50 Connection of the flow monitoring set

6. Read the current gas flow rate from the menu System ▸ Component test in the Flow tab.
7. If the displayed flow rate diverges from the target flow rate by more than ±10 ml/min, find the cause and correct the defect.

   The flow rate shown in the software does not correspond with the flow rate applied in the system. The argon bypass flow rate is not registered. The bypass flow rate is at approx. 15 ml/min. It may, however, vary between 10 and 25 ml/min. This value must be considered when measuring the flow rate.
8. Insert the disposable filter in between the hoses for a second measurement and repeat the reading of the current gas flow rate. The filter serves as a small resistance in the gas flow.
9. If the gas flow determined during the 2nd measurement deviates from the gas flow in the 1st measurement by more than 2 %, search the system for obstructions and leaks.

   If these measures are not successful, contact the customer service department at Analytik Jena.

10.4.3 Servicing the auto-protection assembly

CAUTION

There is a risk of burns on the combustion furnace and on the heated gas transfer line! Only install the auto-protection assembly when the device is cold or allow the device to cool down sufficiently!

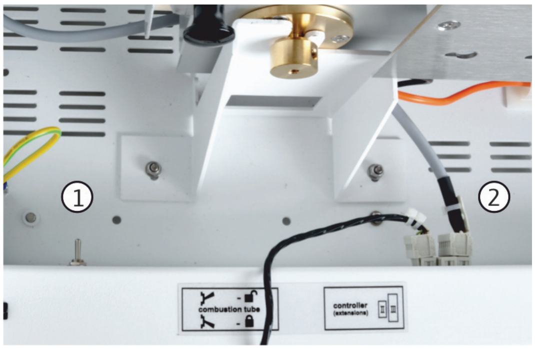
NOTICE

Always switch off all components of the multi X 2500 before electrically connecting them! Connecting or disconnecting electrical contacts may damage the sensitive electronic components of the multi X 2500.

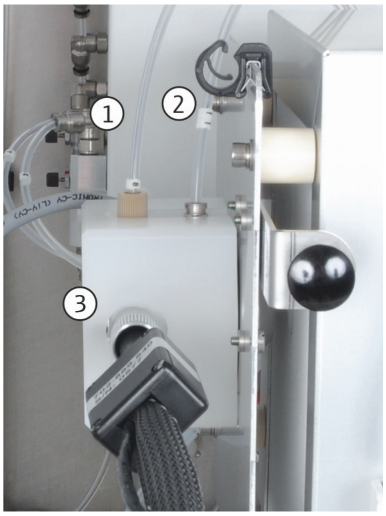
Removing the auto-protection assembly

For the following maintenance work the auto-protection assembly must be removed from the multi X 2500.

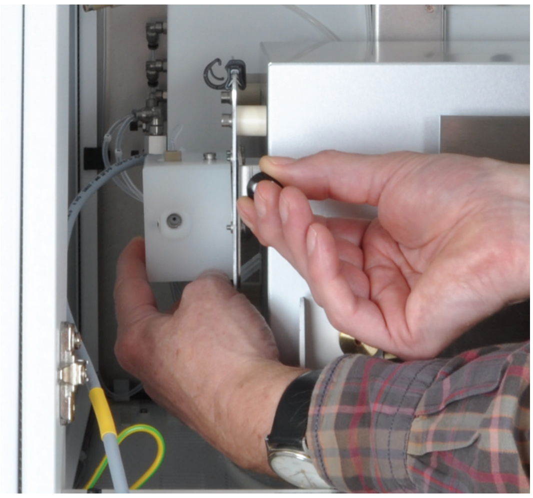
1. Exit the multiWin software and allow the analyzer to cool down.
2. Disconnect the gas transfer line from the sulfuric acid container in the chlorine module.
3. Use the mains switch to switch off the multi X 2500 and the chlorine module and cut the gas supply.
4. If not already done, tilt the combustion furnace into horizontal position.



5. Open the pneumatic seal on the auto-protection assembly. To do this, move the toggle switch (1) up.
6. Pull on the multi-purpose combustion tube to remove it a little from the combustion furnace.
7. Pull the electrical connector for the auto-protection assembly (2) from the connection.



8. Detach hose no. 8 (1) from the connection.
9. Remove hose no. 11 (2). Push down the ring of the plug-in connector to do this.
10. Detach the gas transfer line (3) from the connection at the side.

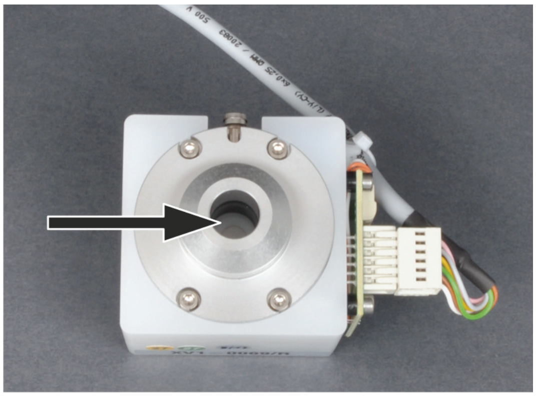


11. Hold the auto-protection assembly with the left hand and pull the knob of the clamping mount with the right hand to open the lock.

    Remove the auto-protection assembly from the bracket on the combustion furnace.

Check/replace filter

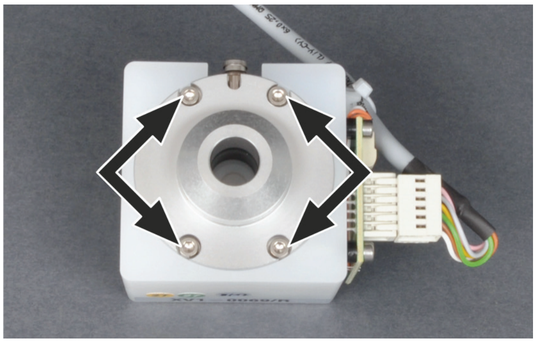
The condition of the filter in the auto-protection assembly must be checked every three months. Proceed as follows:



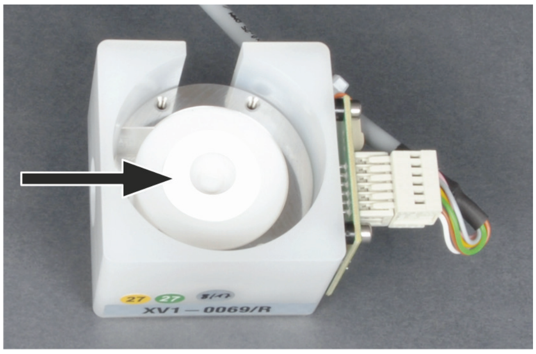
1. Remove the auto-protection assembly.
2. Check the filter for carbonization, soiling or cracks through visual inspection.

   If the filter is OK, re-install the auto-protection assembly.

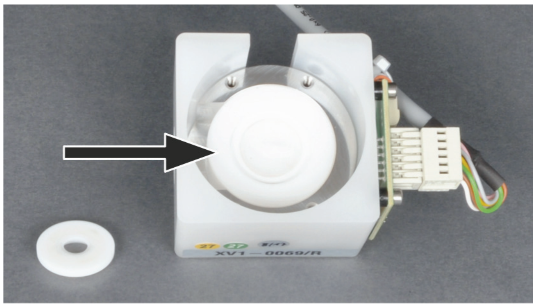
   If the filter needs replacing, follow the remaining instructions.



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



5. Remove the white intermediate ring.



6. Remove the worn filter and insert a new filter.

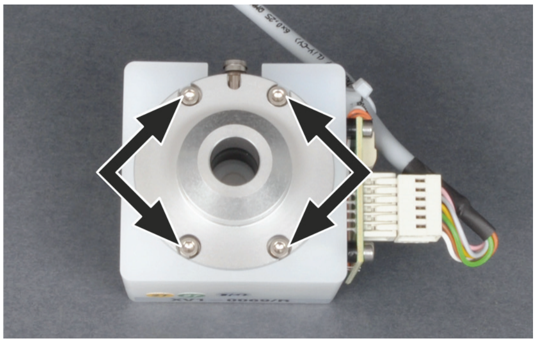
   The installation position of the filter does not matter.

7. Reassemble the auto-protection assembly in reverse order and reinsert it to the multi X 2500.

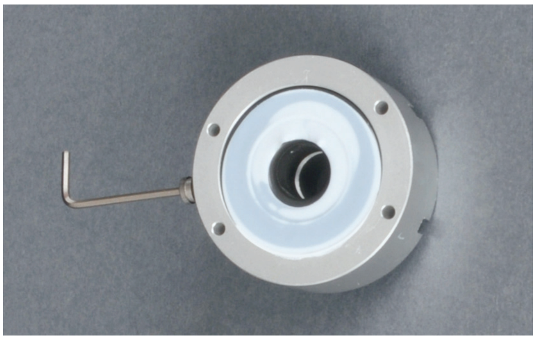
   ✓ The auto-protection assembly is ready for operation again.

Replacing the pneumatic seal:

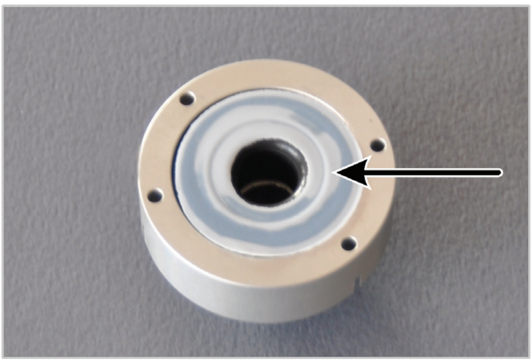
1. Remove the auto-protection assembly.



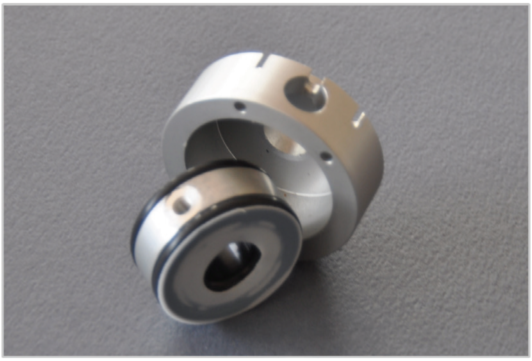
2. Unscrew the 4 screws attaching the pneumatic seal to the auto-protection assembly.



3. Remove the housing with the pneumatic seal from the auto-protection assembly.
4. Use an Allen wrench to unscrew the connection bushing for hose no. 11 from the seal housing. To do this, insert the Allen key into the connection bushing.

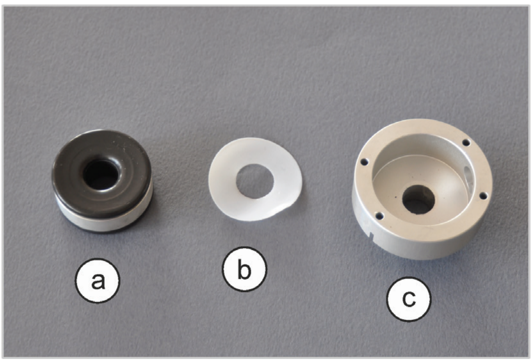


5. Pull the PTFE disk from the seal (see arrow).

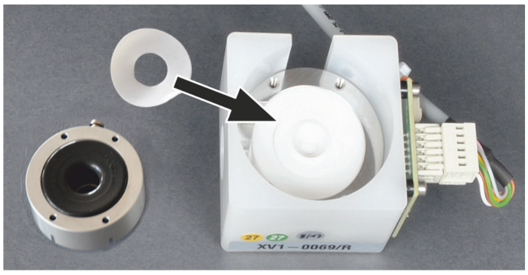


6. Remove the pneumatic seal from the housing.
7. Pull the second PTFE disk from the bottom of the seal.

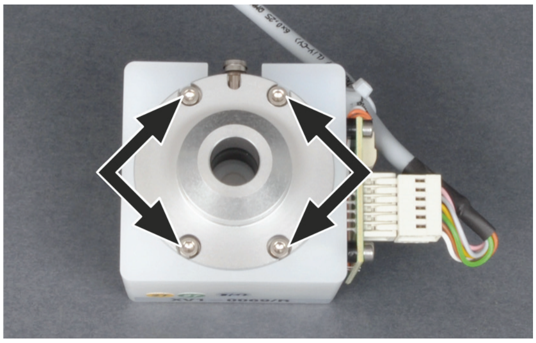
8. Carefully separate the black special seal from the ring.
9. Insert a new special seal into the ring.



10. Place a PTFE disk (b) into the housing (c).
11. Place the pneumatic seal (a) into the housing. The hole in the ring must align with the hole in the housing.
12. Use the Allen wrench to tighten the connection bushing for hose no. 11 (see item 4).



13. Place a second PTFE disk onto the intermediate ring above the filter.



14. Place the pneumatic seal onto the auto-protection assembly and attach it with 4 screws.
    - ✓ The auto-protection assembly can now be reinstalled into the analyzer.

10.4.4 Removing and cleaning the multi-purpose combustion tube

WARNING

Danger of explosion! The hose connections for argon and oxygen on the combustion tube must not be mixed up when reinstalling the assembly.

CAUTION

Risk of burns! Only remove the combustion tube when the device is cold!

NOTICE

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

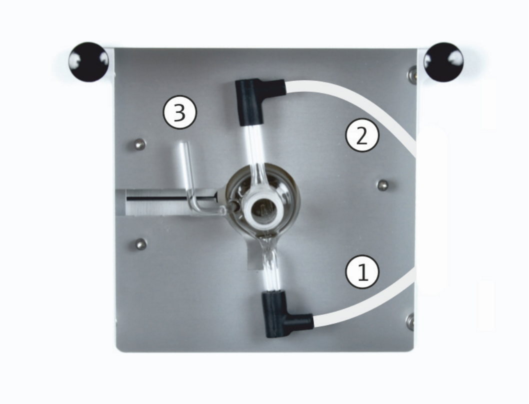
Do not touch the combustions tube with your bare hands during reinstallation. Wear protective gloves. If necessary, clean the combustion tube externally before installing it in the combustion furnace (e.g. by wiping it with moist cellulose).

NOTICE

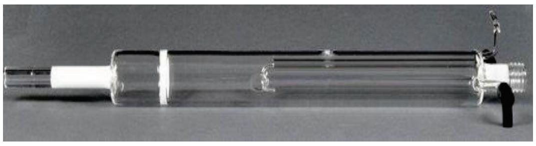
Closing the seal when the combustion tube has not been inserted into the furnace may destroy the pneumatic seal of the auto-protection assembly. Only move the toggle switch down if a combustion tube has been inserted. Alternatively, we recommend to cut the gas supply during maintenance.

1. Exit the multiWin control software and allow the analyzer to cool down.
2. Disconnect the gas transfer line from the sulfuric acid container in the chlorine module.
3. Switch off the multi X 2500 and the chlorine module. Remove the mains plug from the power outlet. Switch off the gas supply.
4. In vertical operating mode, remove the cover at the top.

5. Open the front doors of the analyzer.
6. Open the pneumatic seal to the auto-protection assembly. To do this, move the toggle switch up. This releases the combustion tube.
7. Allow the device to cool down.



8. Pull hose no. 3 (1) and hose no. 4 (2) from the angled adapters at the combustion tube. If used, remove the flame sensor from the connection (3).
9. Carefully remove the combustion tube from the furnace.



10. Check the combustion tube for excessive crystallization, cracks and blow-outs. Only reuse intact combustion tubes.
11. Clean the combustion tube with detergent and a brush or cellulose, if necessary.

    The combustion tube can also be cleaned by burning off deposits in a muffle furnace or with a suitable burner flame.

Refer to "Inserting the multi-purpose combustion tube" S. 67 for information on the reinstallation of the multi-purpose combustion tube.

10.4.5 Changing the sulfuric acid and cleaning the sulfuric acid container

WARNING

Concentrated sulfuric acid can cause severe chemical burns! Wear protective clothing when working at the sulfuric acid container. Observe all specifications in the safety data sheet!

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. As a result, the measured AOX contents will be too low and widely scattered. For this reason, the sulfuric acid must be replaced once per day. High sample throughput may require more frequent replacement.

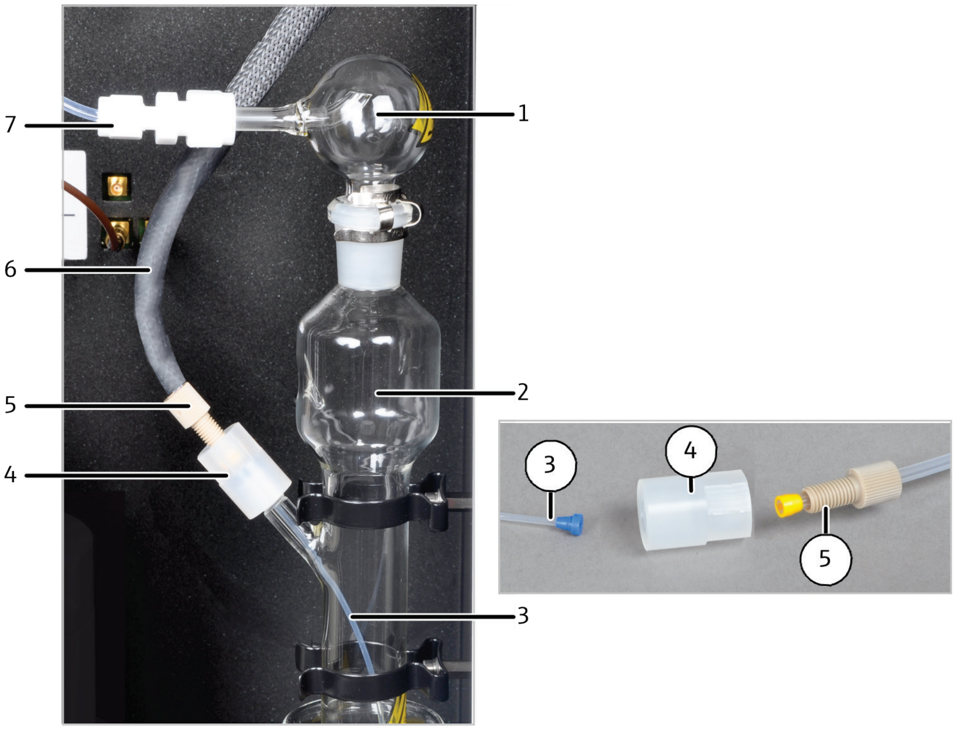


Fig. 51 **Connection transfer line – sulfuric acid container**

1	Safety attachment	5	Banjo bolt
2	Sulfuric acid container	6	Measuring gas transfer to the measuring cell
3	Hose to introduce measuring gas	7	Gas transfer line to measuring cell
4	Connector		

1. Exit the multiWin software. 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.

2. Remove the banjo bolt (5 in Fig. 42) from the connector, separating the heated gas transfer line from the sulfuric acid container.
3. Disconnect the Teflon connection (7 in Fig. 42) and remove the measuring gas hose from the safety attachment.
4. Carefully pull the sulfuric acid container with its remaining components out of the holding clamps toward the top and remove it from the module.
5. A large beaker is suitable for safe transport and storage (e.g., 500 ml).
6. For the "high sensitive" measuring cell: Remove the gas inlet tube from the measuring module with Teflon screw joint and hose.
7. Remove the safety attachment from the sulfuric acid container.

   NOTICE! The bases of the Teflon screw joints remain on the safety attachment, the hose and the gas inlet tube.

8. Unscrew the connectors for the heated gas transfer line from the sulfuric acid container.
9. Remove the thin hose from the container.

   CAUTION! Sulfuric acid residue can still be present on the hose.

10. Empty the sulfuric acid via the top opening. Dispose of the sulfuric acid.
11. Rinse the sulfuric acid container and the safety attachment multiple times with ultrapure water and then rinse with ethanol or methanol.
12. Rinse hose including the Teflon screw joint with ultrapure water and then with ethanol or methanol.
13. Dry the cleaned components, e.g., by blowing it through with an inert gas.
14. Place the sulfuric acid container down somewhere safe and fill it with 20 ml of concentrated sulfuric acid.
15. Install the filled sulfuric acid container in the detection module in reverse order.

    NOTICE! Ensure the proper fit of the conical nipples when connecting the gas transfer line and the PTFE connectors.

10.4.6 Measuring cell maintenance

Since the continuous introduction of hot reaction gases during operation causes electrolyte components to evaporate, it is recommended to change the electrolyte solution once per day for all measuring cells:

1. Remove the used electrolyte.
2. Rinse the measuring cell and the magnetic stirring rod with distilled water and with ethanol.
3. Then, carefully wipe out the measuring cell and wipe the magnetic stirring rod with cellulose to remove any silver chloride deposits.
4. Fill the measuring cell with fresh electrolyte:

Measuring cell “high sensitive”:	approx. 65 ml
Measuring cell "sensitive":	20 ml
Measuring cell "high concentration":	approx. 120 ml

Also observe the following:

- If the system is decommissioned for several days, clean the measuring cell and store it dried.
- Regularly check the casing of the magnetic stirring rod for cracks. Metal ions entering the electrolyte solution can distort the analysis.
- Avoid fluids from entering the stirring/cooling block and the plug-in contacts (risk of short circuit).

10.4.7 Cleaning and storing the electrodes

Refer to the chapter "Clean and store the combined electrode" on page 92 for information on the maintenance of the combined electrode.

Sensor electrode

Observe the following instructions when servicing the sensor electrode:

Note

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 long-term storage (>1 month). Apply scratch protection to the sensor pin. Store the electrode in a dry place.

Reference electrode

CAUTION

Risk of burns! Only install the combustion tube in the cold state!

- 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 and leave the electrode in the filled measuring cell, where it is stored in a moist and dark place.
- For storage longer than 1 month: Close the refill opening. Apply the empty protective cap to the electrode. Store the electrode in a dark, upright position, preferably in the original packaging.

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

Make sure that the opening for the salt bridge of the platinum electrode is not obstructed. Otherwise, the electrode is maintenance-free.

Silver electrode

Wipe the silver surface with cellulose after use. Otherwise, the electrode is maintenance-free. The silver electrode is consumed with time when in operation and must be replaced as required.

10.5 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 vis-cous 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.

Note

Contamination, particles and moisture can damage the Teflon seal of the plunger during assembly. This cau-ses 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.

11 Fault removal

For fault analysis it is possible to record log files. The recording of log files should be enabled in coordination with the Analytik Jena customer service department.

- Enable the recording of the device-specific log files multiWin_Comm.log and multiWin_Flow.log under EXTRAS ▶ CONFIGURATION ▶ ERROR ANALYSIS.
  The recording of the file multiWin_Error.log is always enabled.
- Enable the recording of the log file specific to multiWin multiWin_Exception.log under EXTRAS ▶ CONFIGURATION ▶ ERROR ANALYSIS.
- Use the menu command HELP ▶ ERROR ANALYSIS to create a zip file that can be shared with the customer service.

NOTICE

Always inform the customer service department at Analytik Jena if the fault cannot be remedied. This also applies for the repeated occurrence of individual faults. The zip file that was created must be sent by email to the customer service department at Analytik Jena for the fault diagnosis.

11.1 Troubleshooting according to software messages

NOTICE

Unsuitable USB cables or impermissible extensions often cause problems in the communication between the control module and the PC. Only use the USB cable supplied by Analytik Jena. Extensions of the USB connection are not permitted!

	Error message	
1	**Communication error: No response of firmware!**	
	Cause	**Remedy**
	Basic device not switched on	▪ Switch on basic device
	Basic device not connected to PC	▪ Check connection between basic device and PC
	Incorrect port selected on the PC	▪ Check which port the device is plugged in on the PC ▪ If applicable, select different port in multiWin via the menu EXTRAS ▶ INTERFACE ▪ Initialize

2	**Communication error: Serial interface not available!**	
	Cause	**Remedy**
	Communication problems	■ Disconnect USB connection between basic device and PC and re-connect after approx. 10 s ■ Initialize
3	**Communication error: Serial interface not available!**	
	Cause	**Remedy**
	Communication problems	■ Disconnect USB connection between basic device and PC and re-connect after approx. 10 s ■ Initialize
7	**Operating system error: Not authorized accesses!**	
	Cause	**Remedy**
	Undefined termination of multiWin	■ Exit multiWin ■ Detach USB cable and re-connect after approx. 10 s ■ Restart operating system (PC) ■ Restart firmware (basic device) ■ Restart multiWin
12	**Signal echo received, check interface selection**	
	Cause	**Remedy**
	Incorrect port selected	■ Check port selection
14	**Data transfer interrupted**	
	Cause	**Remedy**
	No data transfer for 10 s	■ Initialize
17	**Wrong interface protocol ID**	
	Cause	**Remedy**
	Fault after update (program versions of firmware multiWin don't match)	■ Update required
20	**Timeout InitEnd**	
	Cause	**Remedy**
	Timeout during initialization	■ Initialize
21	**Timeout StatusBusy**	
	Cause	**Remedy**
	Timeout during operation (device not ready to measure)	■ Acknowledge message ■ Initialize
22	**Timeout End**	
	Cause	**Remedy**
	Timeout when exiting multiWin	■ Acknowledge message ■ Initialize
23	**Timeout StopEnd**	
	Cause	**Remedy**
	Timeout during measurement cancellation	■ Acknowledge message ■ Initialize

50	**Firmware reset**	
	Cause	**Remedy**
	Internal computer (firmware) restarted	■ Acknowledge message ■ Initialize
61 62 64 65 66 67	**Incomplete command from PC** **Command from PC without STX** **Command from PC: CRC error** **Command from PC is invalid command** **Command from PC: Invalid MEASURING command** **Command MTXT from PC is missing**	
	Cause	**Remedy**
	Communication error	■ Acknowledge message ■ Initialize
130	**Cl sensor: no connection**	
	Cause	**Remedy**
	Communication faulty after detector was detected during initialization	■ Acknowledge message ■ Initialize
131	**Cl sensor: wrong command structure**	
	Cause	**Remedy**
	Error in communication with chlorine module	■ Acknowledge message ■ Switch chlorine module on/off ■ Initialize
132	**Cl sensor: Indication error**	
	Cause	**Remedy**
	Indicator value after titration start out of range (measurement cannot start)	■ Acknowledge message ■ Initialize ■ Execution of the end point routine ■ Check status of measuring cell via SYSTEM ▸ COMPONENT TEST ▸ CHLORINE
133	**Cl sensor: wrong cell type**	
	Cause	**Remedy**
	No initialization after cell change	■ Initialize
134	**Cl sensor: wrong status**	
	Cause	**Remedy**
	Communication error	■ Acknowledge message ■ Initialize ■ Check titration cell status via menu item SYSTEM ▸ COMPONENT TEST tab CHLORINE
135	**Cl sensor: wrong version**	
	Cause	**Remedy**
	Transmission error	■ Acknowledge message ■ Initialize ■ Check status of titration cell via menu item

200	**Gasbox: no connection**	
	Cause	**Remedy**
	Communication error	■ Acknowledge message ■ Initialize
201	**Gasbox: error while setting up nominal flow**	
	Cause	**Remedy**
	Communication error	■ Acknowledge message ■ Initialize
202 203 204 205	**Gasbox: conversion error 1** **Gasbox: conversion error 2** **Gasbox: conversion error 3** **Gasbox: conversion error 4**	
	Cause	**Remedy**
	Communication error	■ Acknowledge message ■ Initialize
206	**Gas pressure error**	
	Cause	**Remedy**
	Overpressure in the analysis system due to obstructed gas paths, e.g.: ■ combustion tube (inlet, glass drip) ■ filter in the auto-protection assembly ■ Kinked gas hoses ■ Connectors/couplings clogged ■ Gas inlet to measuring cell ■ Adsorption tube See also user manuals of the system modules.	WARNING! Greatest caution is necessary during an overpressure in the system! Never switch off a device that is subject to overpressure! ■ Do not introduce any samples ■ Do not close multiWin ■ Do not switch off basic device and module ■ Do not switch off the gas supply ■ Manually ventilate device via chlorine module: Manual ventilation: WARNING! Concentrated sulfuric acid! Wear safety clothing. ■ Detach hose connection to measuring cell. ■ Detach gas transfer line from sulfuric acid container and remove sulfuric acid. ■ Detach gas transfer line from auto-protection assembly. After the overpressure has dissipated: ■ Switch off devices, cut gas supply. ■ Search for cause for overpressure and correct the defect.
220	**Sampler: no connection**	
	Cause	**Remedy**
	Communication faulty after sampler was detected during initialization	■ Acknowledge message ■ Initialize
222	**Sample boat: broken**	
	Cause	**Remedy**
	Boat broken during removal from the combustion tube (only if ABD is used)	■ Remove broken boat from the system ■ Initialize

222	**Sampler: wrong syringe size**	
	Cause	**Remedy**
	No syringe inserted	▪ Insert syringe into autoX 112
	Dosing volume in the method greater than inserted syringe	▪ Adjust dosing volume
	The method for dosing liquids is to be enabled and the gripper and solids track are still inserted	▪ Insert sample rack for liquid samples into autoX 112 ▪ Initialize
224	**Sampler: wrong gripper**	
	Cause	**Remedy**
	No gripper inserted	▪ Insert gripper into autoX 112
	The method for solids is to be enabled and the syringe and sample rack for liquid samples are still inserted	▪ Insert solids rack into autoX 112 ▪ Initialize
225	**Sampler: position not reached**	
	Cause	**Remedy**
	autoX 36 – target position is not reached	▪ Check autosampler for mechanical hindrance, remove hindrance if required ▪ Replace rack ▪ Initialize
226	**Sampler: runtime exceeded**	
	Cause	**Remedy**
	Message from autoX 112 sampler saying that the movement is completed takes too long (autoX 112 defective)	▪ Record log files ▪ Report to customer service
230	**ABD: no connection**	
	Cause	**Remedy**
	Communication error after ABD was detected during initialization	▪ Acknowledge message ▪ Initialize
232	**Flame sensor error**	
	Cause	**Remedy**
	Flame sensor calibration failed	▪ Record log files ▪ Report to customer service
260	**Sample handling missing**	
	Cause	**Remedy**
	No sampler module detected	▪ Connect at least one sample introduction module (autoX, auto-injector, ABD, MBD) ▪ Initialize
270	**Autoinjector - syringe: no connection**	
	Cause	**Remedy**
	No communication with auto-injector	▪ Acknowledge message ▪ Initialize
271	**Autoinjector - syringe: runtime exceeded**	
	Cause	**Remedy**
	▪ Communication error with auto-injector ▪ Completion message at dosing end not received	▪ Acknowledge message ▪ Check auto-injector ▪ Initialize

272	**Auto-injector – syringe: incorrect size**	
	Cause	**Remedy**
	Dosing volume and syringe size differ	■ Adjust dosing volume and syringe size
273	**Auto-injector – syringe: not filled properly**	
	Cause	**Remedy**
	Syringe not drawn all the way to the stop	■ Draw syringe completely ■ Insert syringe
274	**Auto-injector: no connection**	
	Cause	**Remedy**
	Auto-injector coupling was not found	■ Check connection ■ Acknowledge message ■ Initialize
300	**Temperature controller: no connection**	
	Cause	**Remedy**
	Communication error	■ Acknowledge message ■ Initialize
304	**Temperature controller: communication error**	
	Cause	**Remedy**
	Temperature cannot be set	■ Acknowledge message ■ Initialize
	Communication error	■ Acknowledge message ■ Initialize

11.2 Equipment faults and analytical problems

11.2.1 General notes

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

If the suggested solutions are not successful, inform the customer service department at Analytik Jena.

11.2.2 Equipment fault

furnace does not heat	
Cause	**Solution**
■ Thermocouple connector not connected	■ Connect thermocouple connector (4 in Fig. 5, p. 20)
■ 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 fuse check the connection b/w basic device and PC
■ Malfunction in the internal electronics	■ Report to customer service

Furnace temperature is outside tolerance limits or target temperature is not reached

Cause	Solution
▪ Temperature controller faulty	▪ Report to customer service
▪ Electronics error	

Process gases (inlet flow) are not present

Cause	Solution
▪ Gas supply not connected	▪ Connecting the gas supply
▪ Primary gas pressure too low	▪ Set primary gas pressure at the delivery point to between 4 and 6 bar
▪ Gas supply leaking	▪ Check gas supply
▪ No method loaded	▪ Load method
▪ Gas box faulty	▪ Report to customer service

Target flow at the outlet to the detector module too low

Cause	Solution
▪ Connection between hose, angled adapter and combustion tube not correct	▪ Check connection and ensure correct fit at the connection points
▪ Pneumatic seal in the auto-protection assembly does not seal combustion tube	▪ Check argon supply, open the pneumatic seal with the toggle switch
▪ Septum sits incorrectly at the inlet of the combustion tube or is leaking	▪ Check position of the septum, insert new septum if necessary
▪ Connection transfer line to the auto-protection assembly leaking	▪ Check connection
▪ In the horizontal operating mode transition coupling tube to ABD leaking	▪ Check sealing of the coupling element check alignment between combustion tube, coupling, ABD, hand-tighten the connection

Gas escapes from pneumatic seal (audible hiss)

Cause	Solution
▪ Screw connection of the gas connection for hose no. 8 is loose	▪ Tighten screw connection and lock nut
▪ Pneumatic seal faulty	▪ Replace pneumatic seal (→ chapter 10.4.3, p. 96)

Auto-protection assembly does not heat up

Cause	Solution
▪ Auto-protection assembly not connected	▪ Plug in the connector (3 in Fig. 6, p. 20)
▪ Heating or temperature controller defective	▪ Inform customer service

Auto-injector is not recognized

Cause	Solution
▪ Auto-injector and autoX 112 are switched on at the same time	▪ Switch off the autosampler autoX 112

11.2.3 Analytical problems

Combustion at the cannula

Cause	Solution
■ Argon and oxygen connection on the multi-purpose combustion tube were mixed up	■ Connect gases correctly (1-2 in Fig. 10, p. 22)

Low results independent of detection

Cause	Solution
■ Metering fault	■ Check metering
■ System leaking	■ Check system tightness (→ chapter 10.4.2, p. 93)
■ Temperature set too low	■ Check temperature configurations in the method
■ Wrong or unsuitable calibration	■ Check calibration, repeat if required
■ Sample loss at or before sample introduction due to vaporization or spillage	■ Close liquid samples Check the sampler routine for solid samples
■ Insufficient post-combustion period	■ Particularly for solids, set a post-combustion time of 120 s in the method
■ Sooting in the system	■ Clean or replace sooted parts

Carryover

Cause	Solution
■ Inadequate rinsing of the sample introduction components	■ Rinse metering syringes prior to sampling
■ Combustion tube insufficiently rinsed for EOX determinations	■ Rinse combustion tube with clean solvent, i.e. blank measurements until values are constant
■ Contamination within the injection port of the combustion tube or within the ABD sluice	■ Replace septum, clean sluice

Scattering measured values

Cause	Solution
■ Metering faulty	■ Check metering
■ Combustion tube contaminated or severely crystallized	■ Clean or replace combustion tube

12 Transport and storage

Observe the safety instructions in chapter "Safety instructions, transport and commissioning", p. 9. Take particular care when transporting the basic device and the chlorine module to prevent damage from impact or vibration.

Use the original packaging for transporting the basic device and its system components (e.g. electrodes). Use individual, fracture-proof packaging for all glass parts.

WARNING

Concentrated sulfuric acid can cause severe chemical burns! Wear appropriate protective clothing when working at the sulfuric acid container. Observe all specifications in the safety data sheet!

12.1 Prepare device for transport (standard version)

1. Exit the multiWin control software and allow the analyzer to cool down.
2. Use the device switch to switch off the basic device and the chlorine module respectively.
3. Cut the gas supply.
4. Disconnect the mains plugs from the power outlets.
5. Detach all connections on the rear panel of the basic device and the rear panel of the chlorine module:
   - Gas connectors
   - Serial connection cable
   - Mains cable
   - Exhaust air hose

In basic device

1. Detach the hose connection to the measuring cell and the connection to the combustion tube (forked clamps) on the sulfuric acid container (vertical).
2. Empty and rinse the sulfuric acid container. Remove the collection tray of the combustion furnace.
3. Remove the combustion tube and the oxygen sluice from the combustion furnace.
4. Remove the combustion furnace (→ "Removing the combustion furnace", p. 116).
5. Detach all hoses from the basic device or put the hose ends into small bags and attach them to the device with adhesive tape.
6. Secure the top cover of the basic device and the maintenance flaps on the right-hand side of the device with adhesive tape.

In the chlorine module

1. Remove the combined electrode from the measuring cell. To do this, detach the hose connection and the electrical connection. Put the electrode into its original packaging.
2. Detach the electrical connection of the measuring cell. Remove the measuring cell from the chlorine module and rinse it.
3. Remove the adsorption tube.
4. Remove the storage container for the combined electrode from the bracket in the door of the chlorine module.
5. Use adhesive tape to fix the extraction hose of the chlorine module.

12.2 Removing and installing the combustion furnace

12.2.1 Removing the combustion furnace

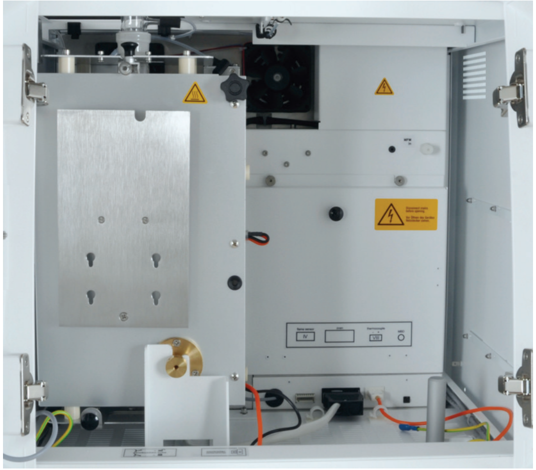
WARNING

Danger of electric shock! Before removing the combustion furnace switch off the multi X 2500 using the mains switch and disconnect the mains plug from the power outlet!

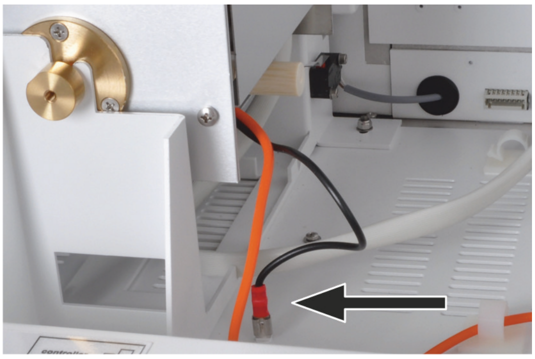
CAUTION

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

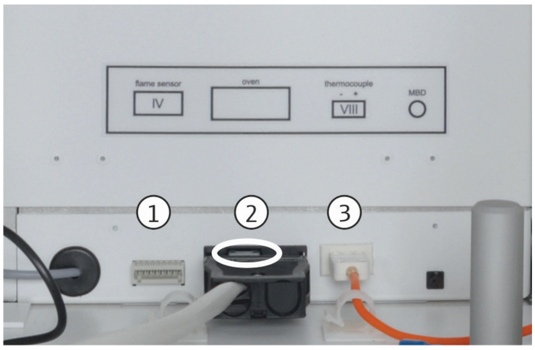
1. Exit the multiWin software. Allow the basic device to cool down.
2. Switch off the multi X 2500 and the chlorine module using the mains switch and pull the mains plugs from the power outlet. Cut the gas supply.
3. Remove the top cover. Open the front doors and remove them from the device.
4. Remove the combustion tube (→ "Removing and cleaning the open combustion tube", p. 88 or "Removing and cleaning the multi-purpose combustion tube", p. 100).



5. Tilt the combustion furnace into vertical position to access the interfaces in the device interior.

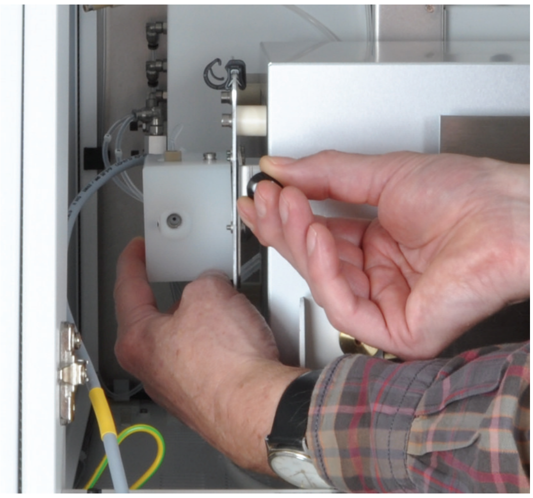


6. Remove the grounding conductor of the combustion furnace from the floor plate connection.

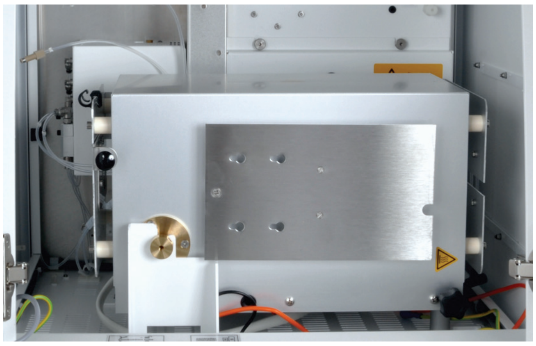


7. Detach the connectors from the slots.
   - Connector of the flame sensor (1)
   - Connector of the combustion furnace (2)
   - Connector of the thermocouple (3)

   To do this, slightly lift the gray lever on the connector of the furnace (2) (see circle).



8. Tilt the combustion furnace into horizontal position.
9. If required, remove the auto-protection assembly from the combustion furnace (→ "Servicing the auto-protection assembly", p. 96).



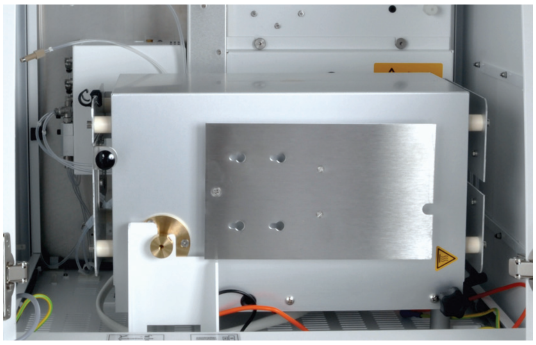
10. Carefully lift the combustion furnace out of tilting mechanism of the multi X 2500.
11. Reinsert the front doors.

12.2.2 Installing the combustion furnace

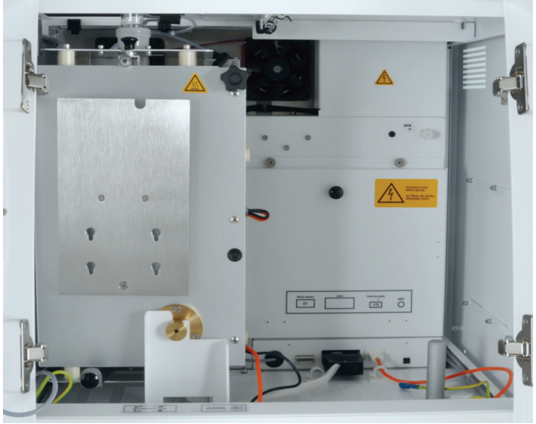
WARNING

Danger of electric shock! Before installing the combustion furnace switch off the multi X 2500 using the mains switch and disconnect the mains plug from the power outlet!

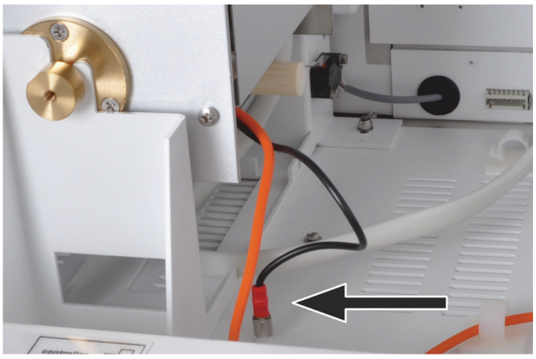
1. Make sure that the multi X 2500 is not connected to the mains.
2. Remove the top cover. Open the doors of the multi X 2500 and remove them from the device.



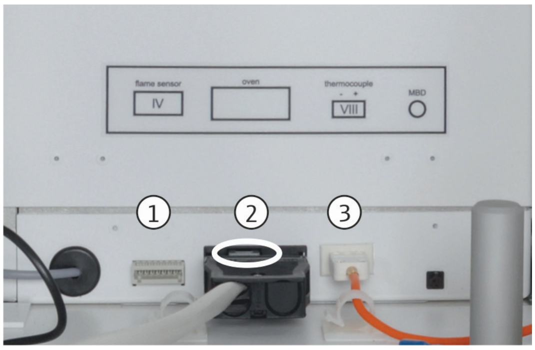
3. Insert the combustion furnace horizontally into the multi X 2500.



4. Tilt the combustion furnace into vertical position to access the interfaces in the device interior.

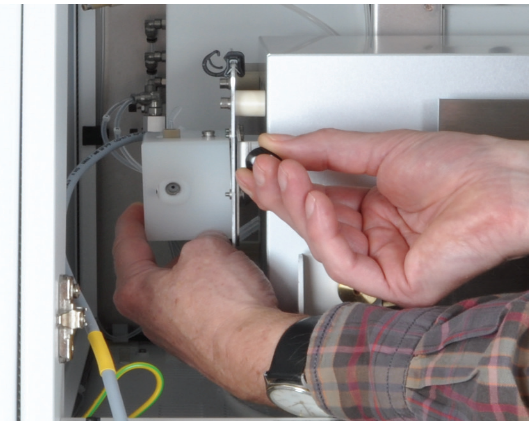


5. Plug in the grounding conductor of the combustion furnace in the floor plate connection.



6. Plug in the connectors to their respective slots:
   - Connector of the flame sensor (1)
   - Connector of the combustion furnace (2)
   - Connector of the thermocouple (3)

   Place the cables for the flame sensor and the thermocouple into the hose clamps.



7. Tilt the combustion furnace into horizontal position.
8. If required, install the auto-protection assembly (→ "Installing the auto-protection assembly", p. 65).
9. Install the combustion tube (→ "Inserting the open combustion tube", p. 55 or "Inserting the multi-purpose combustion tube", p. 67).
10. Place the top cover on top of the device and reinsert the two front doors. Close the front doors.

    ✓ The combustion furnace is now fully installed.

12.3 Prepare device for transport (optional version)

Note

Do not switch off the basic instrument until you have removed the sulfuric acid or until the instrument has cooled down. A vacuum may build up when the device cools down which may draw sulfuric acid into the gas transfer line. As long as the basic device is switched on, the argon safety bypass on the auto-protection assembly prevents this from happening.

If the multi X 2500 is operated with the optional multi-purpose combustion tube, proceed as follows to prepare the device for transport:

1. Exit the multiWin control and analysis software.
2. Remove the gas transfer line from the sulfuric acid container and drain the sulfuric acid (→ "Changing the sulfuric acid and cleaning the sulfuric acid container", p. 101).

WARNING! Sulfuric acid can cause severe chemical burns. The grinding of the sulfuric acid container and the hose which is immersed in the sulfuric acid container may contain residual 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.

3. After the device has cooled down, switch off the analyzer and the chlorine module using the mains switch. Disconnect the mains plugs from the power outlets.
4. Cut the gas supply.
5. Detach all connections on the rear panel of the basic device and the rear panel of the chlorine module:
   - Gas connections
   - Serial connection cable
   - Power cable
   - Exhaust air hose

In basic device

1. After the device has cooled down, detach all hose connections.

   CAUTION! There is a risk of burns at the hot the combustion furnace!
2. Remove the combustion tube.
3. Detach the gas transfer line from the connections and remove it.
4. Remove the auto-protection assembly.
5. Remove the combustion furnace (→ "Removing the combustion furnace", p. 116).
6. Detach all hoses or put the hose ends into small bags and attach them to the basic device with adhesive tape.
7. Secure the top cover of the basic device and the maintenance flaps on the right-hand side of the device with adhesive tape.

In the chlorine module

1. Disconnect the hose connection from the sulfuric acid container to the measuring cell. Remove the safety attachment. Remove the empty sulfuric acid container and rinse it.
2. Detach the electrical connection of the electrodes. Put all electrodes into their original packaging. When doing this, observe the servicing and maintenance instructions for the electrodes (→ "Cleaning and storing the electrodes", p. 103).
3. Remove the measuring cell and rinse it.

   WARNING! The electrolyte solution of the measuring cell "high sensitive" contains concentrated acetic acid. Be careful when emptying the measuring cell!
4. Attach the extraction hose to the chlorine module using adhesive tape.

12.4 Ambient conditions for transport and storage

NOTICE

Environmental influences and condensation can destroy individual components of the multi X 2500! The multi X 2500 must be stored in an air conditioned room. The atmosphere must be free from halogens and hydrocarbons, low in dust and free from aggressive vapors.

If the multi X 2500 is not installed immediately after delivery or not operated over an extended period of time, its individual components must be stored in their original packaging. A suitable desiccant must be added to the packaging and/or the devices to prevent damage from moisture.

Refer to "Technical data" on page 123 for information on temperature and humidity during storage.

13 Disposal

Sulfuric acid	In a first step, carefully dilute the used desiccant with water before neutralizing it with sodium hydroxide. Observe the corresponding hazard statements and safety advice! The neutralized waste must be disposed of professionally in accordance with the legal disposal regulations.
Electrolyte solution	Carefully neutralize the electrolyte solution before collecting it as silver salt residue in containers. Such residue must be correctly disposed of or recycled in accordance with the statutory requirements.
Adsorption agent	The active carbon used for the adsorption of acetic acid vapor must be disposed of in accordance with legal regulations.
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.
Analyzer	At the end of its service life, the multi X 2500 and all its electronic components must be disposed of as electronic waste in accordance with the applicable regulations.

14 Specification

14.1 Technical data

General characteristics

Designation/type	Analyzer multi X 2500
Dimensions of the basic device (W x H x D)	approx. 510 x 470 x 560 mm
Dimensions of the chlorine module (W x H x D)	approx. 300 x 470 x 560 mm
Weight of the basic device	approx. 25 kg
Weight of the chlorine module	approx. 12 kg

Procedural data

Digestion principle	Oxidative combustion
Digestion temperature	700 to 1100 °C
Measuring gas drying	Concentrated sulfuric acid
Detection principle	Micro-coulometric end point titration (argentometry)
Operating range of the wide-range coulometer	3
Temperature control for the measuring cell	Integrated cooling
Stirring mechanism of the measuring cell	Integrated magnetic stirring rod, fixed number of revolutions

Measuring methods

Vertical	AOX
Horizontal (as an option)	AOX, EOX, TX
Vertical (as an option)	EOX, POX, TOC

Vertical sample introduction

AOX	Direct injection of the loaded activated carbon in the quartz container or ejection of the loaded activated carbon from the container in the open combustion tube
EOX	Direct injection of the extract into the multi-purpose combustion tube via injection port with septum
POX	After purging and transfer in carrier gas stream via special sluice for the open combustion tube
TOC	Direct injection of the aqueous samples into the TOC combustion tube via injection port with septum

Horizontal sample feed

AOX	Transfer of the loaded active carbon with or without quartz container in the quartz glass boat into the multi-purpose combustion tube
EOX	Injection of the extract via injection port with septum or transfer of the liquid samples with ABD into the multi-purpose combustion tube
TX	Direct transfer of solid samples in quartz glass boats into the multi-purpose combustion tube

Measuring cell "high sensitive"

Measurement mode	Potentiometry
Measuring range	0.01 to 10 µg Cl
Generator current	100 µA
Electrolyte volume	65 ml

Measuring cell "sensitive"

Measurement mode	Bi-amperometry
Measuring range	1 to 100 µg Cl
Generator current	1 mA
Electrolyte volume	20 ml

Measuring cell "high concentration"

Measurement mode	Bi-amperometry
Measuring range	10 to 1000 µg Cl
Generator current	10 mA
Electrolyte volume	120 ml

Gas supply

Option 1	Oxygen, 4.5 (or better)
For pure AOX systems and combined AOX/TOC systems:	
Option 2	Synthetic air (from a compressed gas cylinder) Free of hydrocarbons, free of CO_2
Option 3	Purified compressed air (via TOC gas generator) CO_2 < 1 ppm, carbohydrates (as CH_4) < 0.5 ppm Primary pressure: 4 to 6 bar Gas consumption (parameter-dependent): Max. 42 l/h in vertical operation mode Max. 24 l/h in horizontal operation mode
Argon	For AOX horizontal, EOX, POX, TX 4.6 (free from halogens and hydrocarbons) Primary pressure: 4 to 6 bar Gas consumption (method dependent): In vertical operating mode 15 l/h max In horizontal operating mode 10 l/h max

Electrical variables

Power supply	110 to 240 V AC, 50/60 Hz
Fuse for basic device	T 10 A H (2x)
Fuse for chlorine module	T 4.0 A H (2x)
Only use original fuses from Analytik Jena!	
Typical average power consumption of the basic device	1000 VA
Typical average power consumption of the Cl module	100 VA
PC interface	USB

Ambient conditions during operation

Temperature	20 to 35 °C
Humidity	90 % max at 30 °C
Air pressure	0.7 bar to 1.06 bar

Max. permissible altitude	2000 m

Ambient conditions for storage

Temperature	15 to 55 °C
Humidity	10 to 30 % (use desiccant)

14.2 Guidelines and standards

Safety class and safety type

The analyzer has a protection class of I.

The housing has safety type IP 20.

Device safety

The analyzer complies with the safety standards

- EN 61010-1
- EN 61010-2-081
- EN 61010-2-010
- EN 61010-2-51

EMC compatibility

The analyzer has been checked for interference emission and noise immunity.

It meets the requirements for interference emission of

- EN 61326-1 (EN 55011 group 1, class B)

It meets the requirements for noise immunity of

- EN 61326-1 (requirements for use in a basic EMC environment)

Environmental compatibility

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

- ISO 9022-3
- ISO 9022-2

EU directives

The analyzer has been 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 as far as technical safety is concerned. To maintain this condition and to ensure safe operation, the operator must strictly observe the safety and operating instructions contained in this operating manual. For accessories which have also been supplied, and system components from other manufacturers, their operating instructions should be referred to.

Guidelines for China

The device contains controlled substances (according to the guideline "Management Methods for the Restriction of the Use of Hazardous Substances in Electrical and Electronic Products"). Analytik Jena guarantees that the substances will not leak within the next 25 years and, therefore, will not cause any environmental danger or health risk within this period of time if the device is used as intended.