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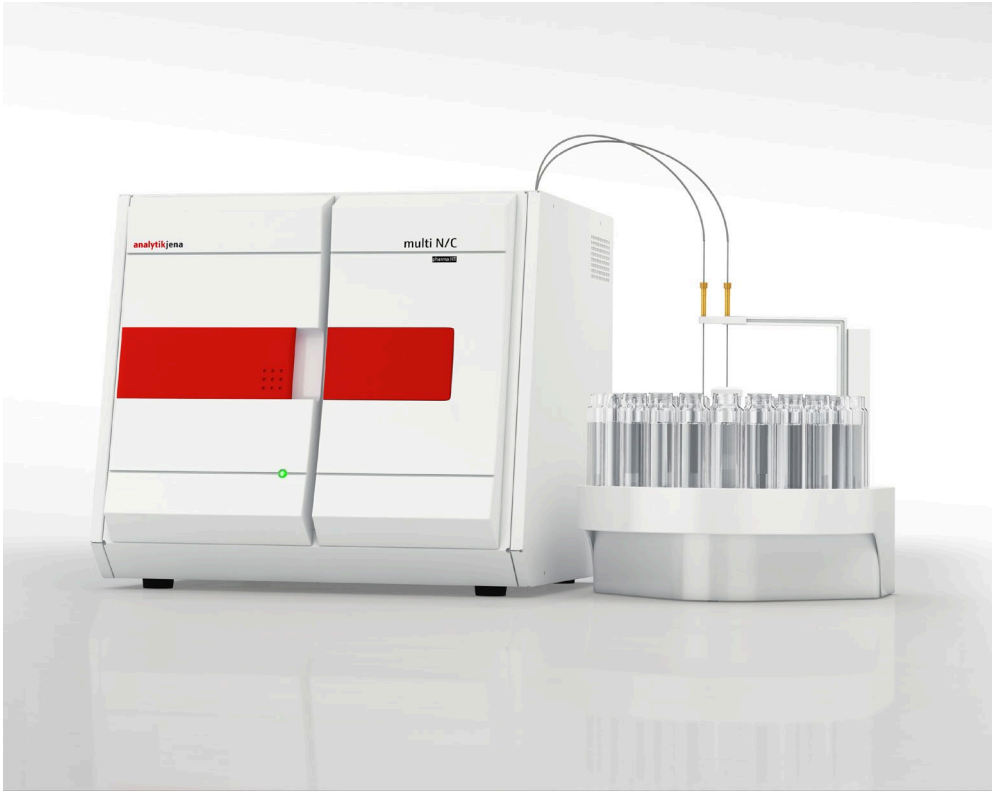
Operating Manual

multi N/C pharma UV

TOC Analyzer



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1 Basic Information

1.1 User manual notes

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

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

Conventions

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

Warnings are marked by a warning triangle 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:

- Terms used in the program are identified with SMALL CAPS (e.g., Menu FILE).
- Buttons are identified by square brackets (e.g., [OK] button)
- Menu items are separated by arrows (e.g. FILE ▸ OPEN)

Symbols and signal words

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

WARNING

Indicates a potentially hazardous situation which might cause fatal or very serious injuries (deformations).

CAUTION

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

ATTENTION

Indicates potential damage to equipment or the environment.

1.2 Intended Use

The analyzer multi N/C pharma UV is a device for determining the parameters TC, TOC, NPOC and TIC in aqueous samples through the wet chemical digestion using UV radiation and peroxodisulfate in accordance with the national and international standards.

The analyzer is intended in particular for the detection of the parameters mentioned in ultrapure water and water for pharmaceutical purposes.

The device must only be used for the methods described in this user manual to determine the total carbon content in aqueous samples. Any other use is not as intended! Only the operator is liable for any damages that result from this.

Improper use can endanger the user or third parties or damage the device. In particular it is prohibited to use the analyzer to analyze flammable liquids or substances that could contaminate the analyzer.

The device must only be used with nitrogen or argon as carrier gases. Oxygen or synthetic air may not be used as carrier gas. The UV radiation would generate ozone due to the dissociation of molecular oxygen.

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

1.3 Warranty and liability

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

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

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

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

2 Safety instructions

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

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

2.1 Safety markings on the analyzer

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

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

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

Fire, naked flame and smoking prohibited!

Warning against dangerous electrical voltage

Warning against hazardous substances

Warning against hot surface

Warning against corrosive substances

Warning mercury

The device contains restricted substances (according to directive SJ/T 11363-2011). Analytik Jena GmbH guarantees, that those hazardous substances may not leak out during the next 25 years when the device is used in accordance with its intended purpose.

2.2 Technical condition

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

The following has to be observed:

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

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

2.3 Requirements for the operating personnel

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

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

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

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

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

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

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

2.4 Safety instructions, transport and installation

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

The following has to be observed:

- Insufficiently secured components pose a risk of injury! During transport the components of the equipment must be secured in accordance with the instructions in the user manual.
- Only transport the analyzer in its original packaging! Ensure that the transport protections have been fitted and the analyzer is completely empty.
- To prevent damaged to health the following must be observed when moving the analyzer in the laboratory (lifting and carrying):
  - For reasons of safety 2 persons are required to transport the analyzer and must position themselves on both sides of the equipment.
  - Because the analyzer does not feature any handles, firmly grip the device from the bottom and make sure prior to simultaneous lifting the device that the sensitive components at the front are protected by the closed doors.
  - The guide values and statutory limits for lifting and carrying loads without auxiliary equipment must be observed and adhered to.

2.5 Safety instructions - operation

2.5.1 General

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

The following has to be observed:

- The analyzer must only be operated if all protective equipment (e.g. covers, drip pans for chemicals and doors) are present, properly installed and fully operational.
- The sound condition of the protection and safety equipment must be checked regularly. Any defects must be corrected as soon as they occur.
- Protective and safety equipment must never be removed, modified or decommissioned during operation.
- Free access to the power switch on the back of the enclosure has to be ensured during operation.
- The ventilation equipment on the multi N/C pharma UV and the extension modules must be in good working condition. Covered vents or ventilation slits etc. may cause the device to break down or may cause damage to it.
- Keep all combustible materials away from the analyzer.

2.5.2 Safety instructions - Protection against explosion and fire

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

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

2.5.3 Safety instructions - electrical equipment

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

The following has to be observed:

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

2.5.4 Safety instructions for compressed gas containers and systems

The carrier gas (nitrogen/argon) is obtained from compressed gas containers or local compressed gas systems. The required purity of the carrier gas must be ensured (→ see section "Technical data" p. 104)!

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

The following has to be observed:

- For gas cylinder or gas plant operation, the safety instructions and guidelines which are valid at the operating location must be strictly complied with.
- High pressure hoses and pressure reducers may only be used for the assigned gases.
- All pipes, hoses and screw connections must be checked regularly for leaks and externally visible damage. Leaks and damaged must be repaired without delay.
- Prior to inspections, service and repairs the valves must be closed and the analyzer vented!

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

2.5.5 Safety notes on UV radiation

- The protection of the user against UVC radiation is ensured by the protective glass before the UV reactor. The protective glass must not be removed during operation.
- Manipulations at the protective glass are not permitted!
- Avoid looking into the protective glass for longer periods of time to protect your eyesight.
- No oxygen or synthetic air may be used as carrier gas! The UVC radiation of the UV reactor would generate ozone due to dissociation of molecular oxygen. Ozone can damage the mucous membranes.

2.5.6 Handling of auxiliary and operating materials

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

When handling dangerous substances local safety codes and guidelines must be observed.

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

The following has to be observed:

- The relevant regulations and the notes in the EC safety data sheets of the manufacturers have to be observed and complied with regards to storage, handing, use and disposal for all auxiliary and operation materials used during operation or maintenance of the analyzer.
- Auxiliary and operation materials may never be placed in containers or vessels for food. The approved containers for the relevant material are to be used and these have to be labelled accordingly. The notes on the labels have to be observed!
- Protective goggles and rubber gloves have to be worn when handing reagents. The notes on the labels have to be observed.
- The regulations and notes on the safety data sheets for the handling of orthophosphoric acid (H_3PO_4), sulfuric acid (H_2SO_4) and sodium peroxodisulfate ($Na_2S_2O_8$) must be observed!
- Biological samples have to be handled according to local guidelines regarding the handling of infectious material.
- Caution when handing quartz glass and glass parts. Risk of broken glass and therefore risk of injury!

- Auxiliary and operating materials as well as their containers may not be disposed in domestic waste or enter the sewage system or the soil. The applicable regulations for disposal of these materials must be meticulously observed.
- The operator is responsible for ensuring that waste materials are disposed of in an environmentally responsible manner and according to local regulations.
- Ensure good room ventilation in working rooms.

2.5.7 Safety instructions - service and repair

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

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

The following has to be observed:

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

2.6 Behavior during emergencies

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

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

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

3 Function and setup

3.1 System design

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

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

All components of the analyzer to be operated or serviced by the user can be reached via 2 doors at the front.

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

- UV reactor with control gear
- Components for sample preparation
- Hose system
- Components for measuring gas drying and cleaning
- NDIR detector
- Accessories
- Electronic component
- Indicator and control elements, connectors

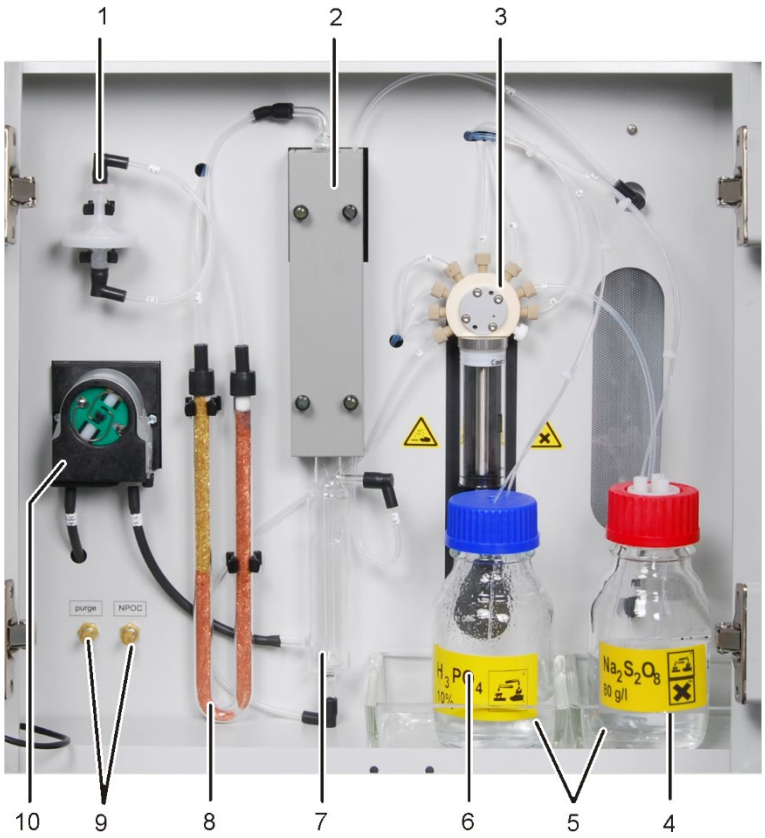


Fig. 1 Front view (doors open)

1 Water traps
2 Cooling block
3 Syringe pump with 9 port valve
4 Reagent bottle for $Na_2S_2O_8$
5 Drip trays
6 Reagent bottle for H_3PO_4
7 TIC condensate container
8 Halogen trap
9 Needle valve to adjust the gas flow
10 Condensate pump

3.1.1 UV reactor with control gear

The analyzer multi N/C pharma UV features a specially developed UV reactor with integrated UV radiation source from quartz glass. The reactor directly surrounds the UV radiation source. Wavelengths of 185 nm and 254 nm are used. This results in a very efficient and high radiation density and thus a high digestion capability|

The reactor has two inlets and one outlet. One inlet is connected to the syringe pump for feeding the sample and reagent. The carrier gas supply takes place via the second inlet. At the top outlet of the reactor the measuring gas is routed via hose connections to the TIC condensate container.

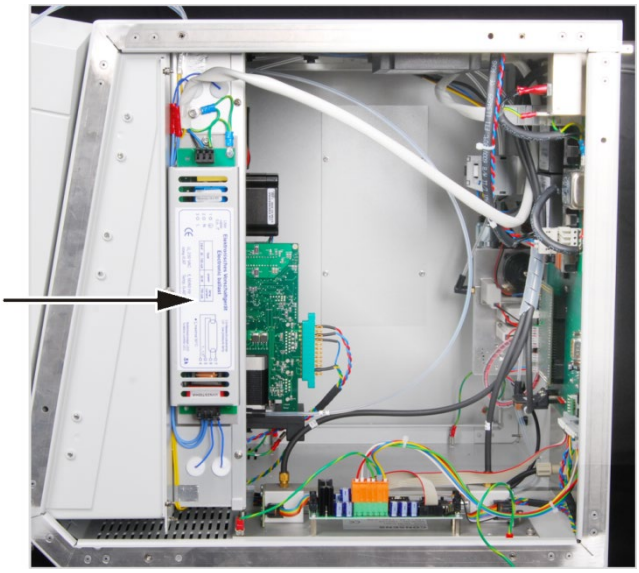
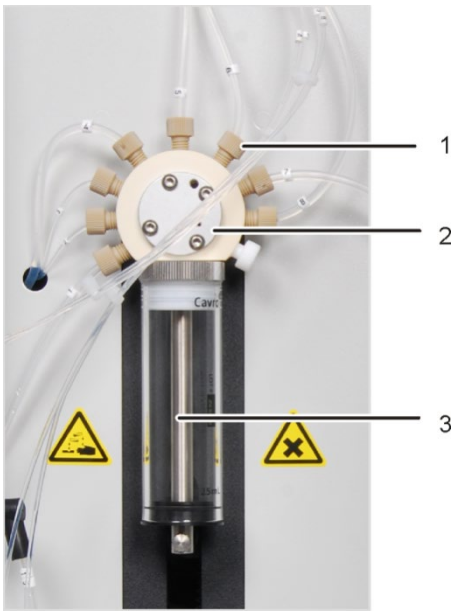


Fig. 2 UV reactor with control gear (right-hand side wall open)

3.1.2 Components for sample preparation

Sample feeding in the analyzer multi N/C pharma UV takes place via flow injection over a syringe pump with 9-port valve. The injection volume is 0.05 to 20 ml. For sample volumes ≤ 1.5 ml system water is additionally entered into the reactor with each metering.

The hose connections are attached to the 9 port valve using Fingertight screw connections. The syringe body is made of glass and replaceable.



1 Fingertight connection
2 9-port valve
3 Metering syringe

Fig. 3 Syringe pump with 9 port valve

The hoses at the 9-port valve are labelled and connected to the following components:

Hose no.	Connection to the component/accessory
1	TIC condensate container
2	Reagent bottle for phosphoric acid H_3PO_4
3	Reagent bottle for sodium peroxodisulfate $Na_2S_2O_8$
4	Waste disposal
5	Ultra-pure water bottle
6	Sample
7	UV reactor
8	Waste disposal
9	not allocated

3.1.3 Hose system

Hose diagram

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

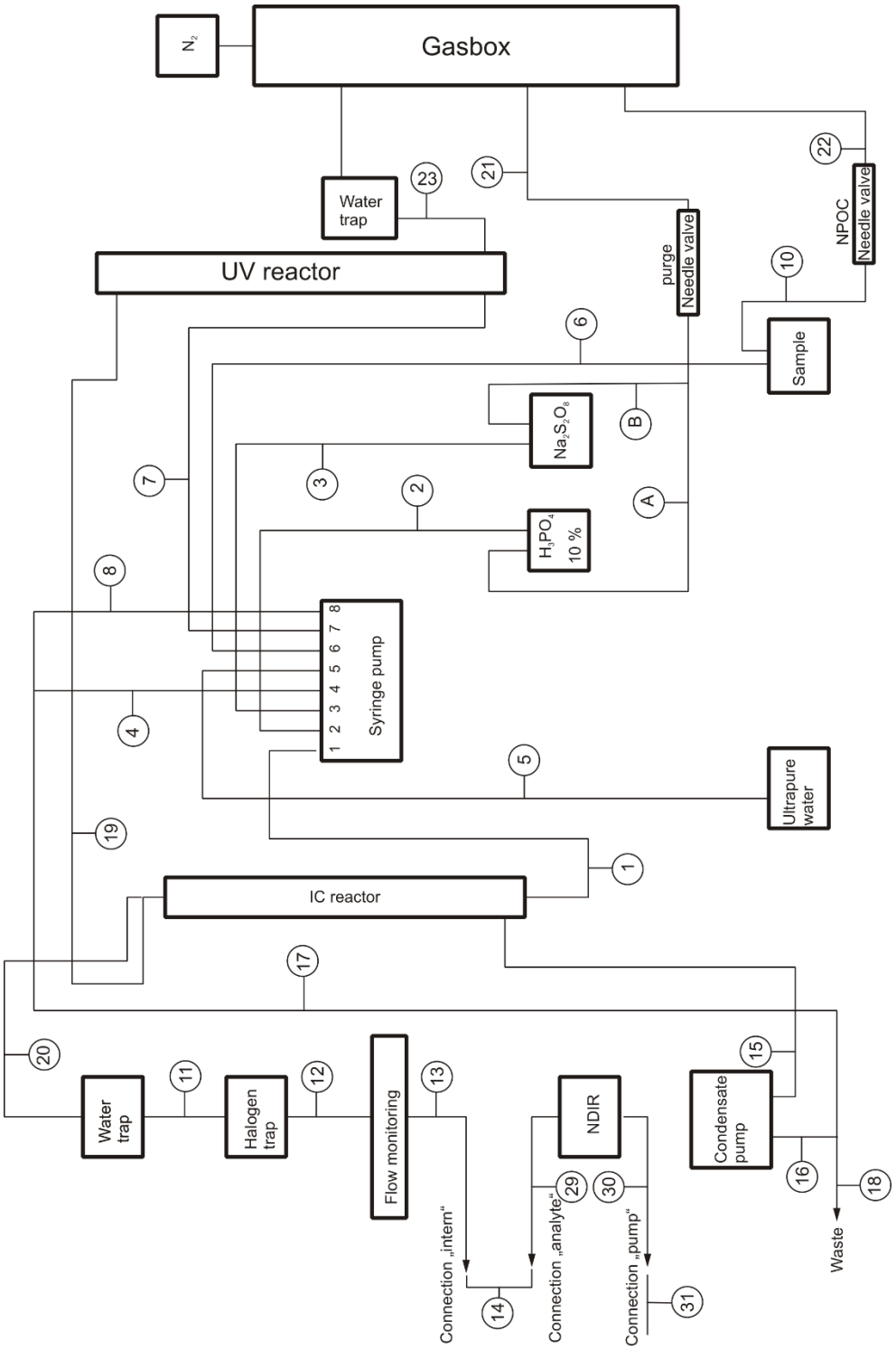


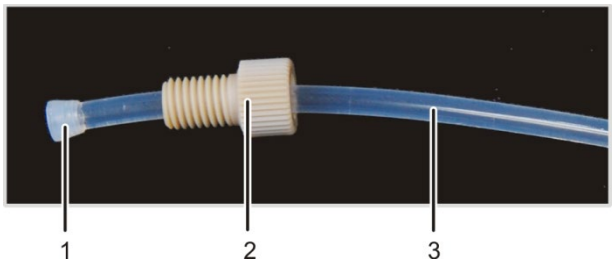
Fig. 4 Hose diagram

Connection method

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

Fig. 5 Different FAST connector designs

In addition, so-called Fingertight screw connections are used in the analyzer multi N/C pharma UV. These flangeless fittings consist of a conical nipple and a banjo bolt. These hose connections seal purely by tightening the plastic banjo bolt finger-tight.



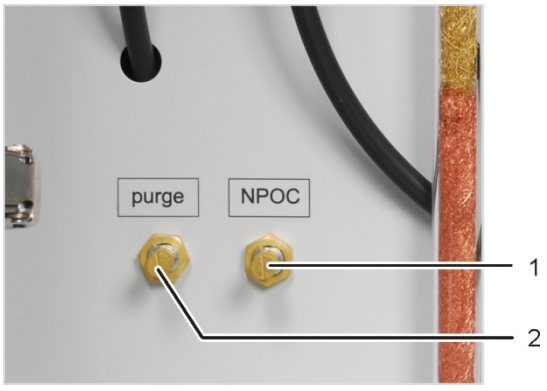
1 Conical nipple
2 Banjo bolt
3 hose

Fig. 6 Fingertight screw connection

Components for flow adjustment

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

The adjustment of the NPOC purge flow and the adjustment of the purge flow for reagents take place via needle valves below the hose pump. The NPOC purge flow is measured using an MFM and displayed in the control and analysis software in the window SYSTEM STATE.



1 Needle valve to adjust the NPOC purge flow
2 Needle valve to adjust the purge flow for reagents

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

Condensate pump

Via the condensate pump the condensate or the waste solution from TIC detection are automatically pumped off after each measurement. The condensate pump is to the left of the halogen trap.

Fig. 8 Condensate pump

3.1.4 Components for gas drying and cleaning

TIC condensation module

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

The TIC condensate container has four connections. One connection at the top connects the TIC condensate container to the UV reactor via a hose supplying the wet measuring gas/carrier gas mixture. The gas is routed downwards on the inside and exits via a glass drip. The integrated glass drip ensures the effective purging of the generated CO_2.

The measuring gas is dried by freezing in the cooling block. A Peltier element provides cooling. The dry measuring gas is routed via the other connection out of the TIC condensate container.

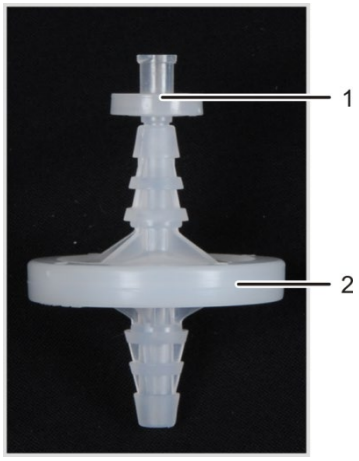
Another hose connection supplies the samples and reagents via the bottom connection into the TIC condensate container. The waste from the TIC condensate container is routed over the fourth connection via the condensate pump to the disposal.

1 Cooling block
2 TIC condensate container

Fig. 9 TIC condensation module

Water traps

The analyzer multi N/C pharma UV contains water traps to remove interfering components from the measuring gas and to protect the detector and the gasbox. The water traps are installed in the gas path after the cooling block and between the gasbox and the UV module. The water traps consist each of a larger water trap (TC pre-filter) that retains aerosol and a smaller water trap (disposable retention filter) retaining rising water.



1 Disposable retention filter
2 TC Pre-filter

Fig. 10 Water traps

Halogen trap

The analyzer multi N/C pharma UV contains a halogen trap (U tube) after the TIC condensate container and the water traps for the greatest possible removal of interfering components in the measuring gas and to protect the detector. The U-tube is filled with special copper wool and brass wool. The filling of the halogen trap has to be renewed at the latest once half of the copper wool or the brass wool is discolored.

Fig. 11 Halogen trap

3.1.5 NDIR detector

The NDIR detector (non-dispersive infrared absorption detector) is on the rear right side wall of the analyzer.

Gases with molecules from different atoms have specific absorption bands in the infrared wavelength range. When a light beam is sent through an arrangement of cells which contains gases in the active IR range, these gas components absorb the characteristic wave lengths with their proportional share of the total radiation according to their concentration in the gas mixture.

The radiation receivers inserted in the used NDIR detector is selective for the CO_2.

Measurements using the VITA method

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

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

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

3.1.6 Indicator and control elements, connectors

LED displays

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

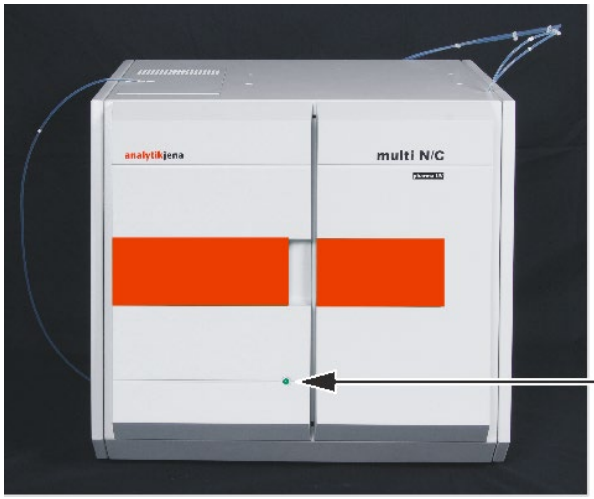


Fig. 12 Diode to indicate readiness for operation

Main switch, interfaces, gas connections

Main switch, mains connection, equipment fuse, media connections (gases and waste) and the interfaces for connecting the PC and the accessories are at the back of the multi N/C pharma UV. A diagram at the center of the backplate explains the different connections.

1 Holder for mains fuse "FUSE"
2 Main switch to switch the analyzer on and off "power switch"
3 Mains connection "main plug"
4 Gas connection "analyte"
5 Gas connection "pump"
6 Connection "internal"
7 Connection for inert gas "N_2 nitrogen"
8 Connection of the neutral conductor at the sampler
9 Waste connection "waste"
10 RS 232 interface for HT module "HT"
11 RS 232 interface for the sampler "sampler"
12 USB port for PC

The connections "analyte" and "internal" are linked via a hose bridge

Fig. 13 Connections on the rear of the device

3.1.7 Reagent accessories

The following accessories are required for measurements with the analyzer:

- connection cables, connection hoses
- suitable waste container or drainage
- reagent bottle with drip pan for phosphoric acid H_3PO_4, 250 ml
- reagent bottle for sodium peroxodisulfate $Na_2S_2O_8$ acidified with sulfuric acid H_2SO_4 for the digestion of carbon compounds up to CO_2, 250 ml
- ultrapure water bottle, 2.5 l

The reagent bottles must be positioned in the drip pans behind the right-hand door. The reagent bottles must be labeled with a safety symbol and the designation of the content.

3.1.8 Autosampler for the analyzer

Four different autosamplers are available for the analyzer:

- AS vario with various tray sizes
- AS 21 for 21 samples
- AS 10 for 10 samples
- EPA sampler with piercing function

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

3.2 Principle of operation

The analyzer multi N/C pharma UV is a compact and powerful device to determine the total carbon content in aqueous samples.

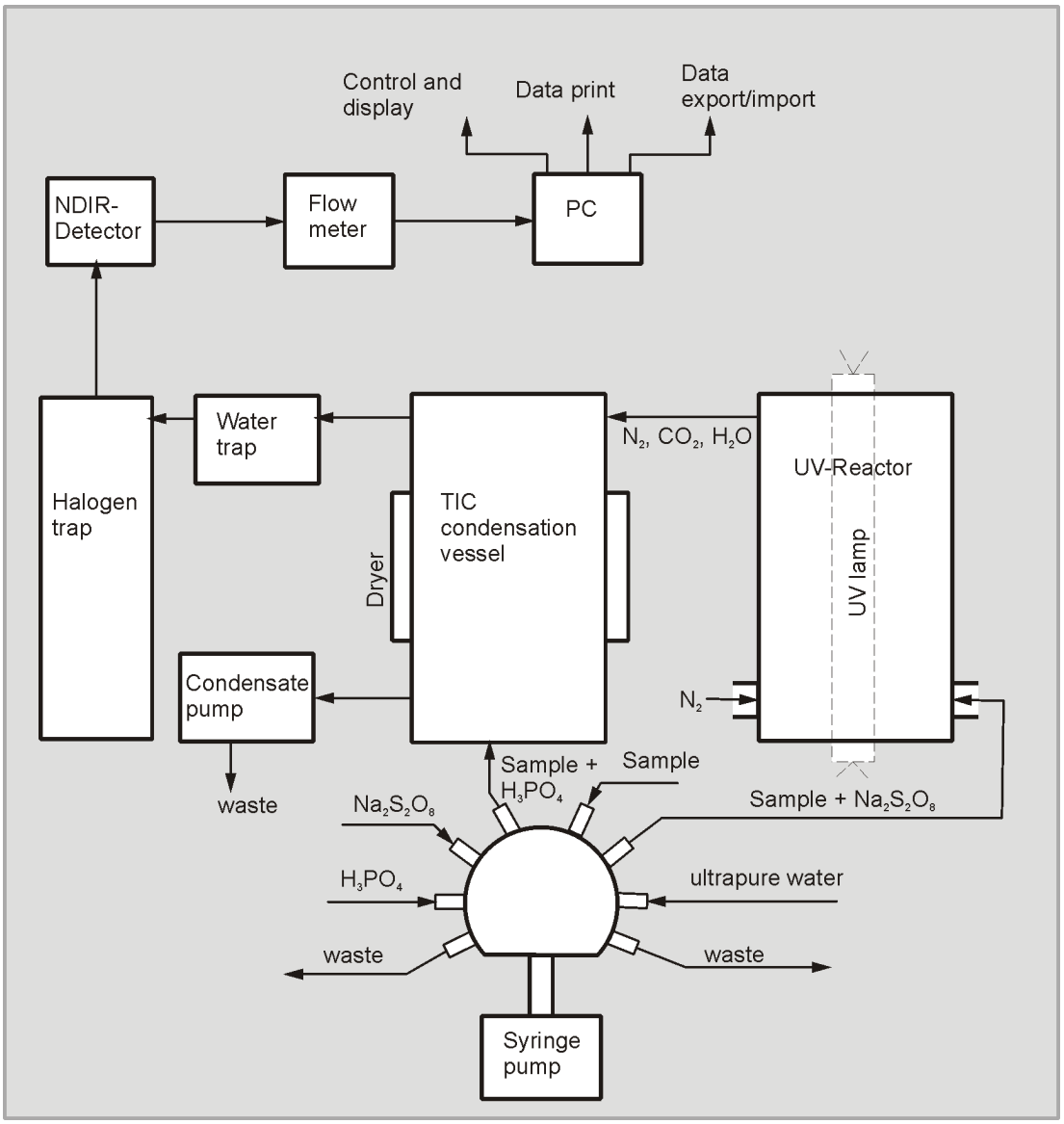


Fig. 14 Principle of operation

The digestion is wet chemical via UV persulfate oxidation or via UV oxidation only. Acidic persulfate solution is added to the sample aliquot and it is radiated with UV radiation of 185 nm /254 nm (UVC) wavelengths. At temperatures of up to 80 °C any carbon compounds contained are broken down up to CO_2. The digestion of the inorganic carbon takes place using H_3PO_4 in the TIC reactor from an additional sample aliquot.

$$R - H \overset{hv}{\rightarrow} CO_2 + H_2O \qquad (1)$$

R-H carbonic organic substance
hv energy

The generated CO_2 is expelled using inert gas (N_2/Ar). After drying and removal of corrosive acting gases, the analyte gas CO2 is added to the NDIR detector.

The concentration of CO_2 is detected several times in one second. An integer over time is calculated from this signal sequence. The integer is proportional to the concentration of the carbon in the measurement agent. Afterwards, the calculation of

the carbon content in the sample is performed via a previously determined calibration function.

3.3 Measuring method

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

- TC - **T**otal **C**arbon
- TOC - **T**otal **O**rganic **C**arbon
- TIC - **T**otal **I**norganic **C**arbon
- NPOC - **N**on **P**urgeable **O**rganic **C**arbon
- DOC - dissolved organic carbon (corresponds to TOC after sample filtration through 0.45 µm filter) (**D**issolved **O**rganic **C**arbon)

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

3.3.1 TC analysis

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

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

3.3.2 TOC analysis

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

The TOC determination in the analyzer is performed according to the differential method, which can be described with the following equation (2).

$$TOC = TC - TIC \qquad (2)$$

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

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

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

3.3.3 TIC analysis

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

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

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

3.3.4 NPOC analysis

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

The sample is acidified to pH 2 outside of the analyzer with H_2SO_4 (c = 2 mol/l) and the resulting CO_2 is purged. Afterwards the remaining carbon from the sample prepared in this manner is digested via the UV reactor and detected in the detector.

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

3.3.5 NPOC analysis according to the NPOC plus method

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

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

As with the NPOC analysis the sample is acidified to pH 2 with H_2SO_4 (c = 2 mol/l) outside the analyzer. Immediately before the analysis of the sample the greater part of the carbon dioxide generated is purged externally. Afterwards the remaining organic carbon (TOC) from the sample prepared in this manner is determined using the differential method.

The TIC value determined using this method is only a calculated variable and of no analytical relevance.

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

3.4 Calibration

3.4.1 Calibration strategies

Single point calibration

For most applications a single point calibration is permitted for the multi N/C pharma UV – the blank value of the device is low and the NDIR detector linear.

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

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

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

Multiple point calibration with constant concentration

For some applications a multiple point calibration with **variable metering volumes** and **constant concentration** can also be used.

A standard solution for the range to be calibrated is prepared measured for different volumes in accordance with the settings in the selected method. The lowest volume of 1.6 ml must not be fallen below.

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

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

Especially for measurements in the lower concentration range (< 1 mg/l) a multiple point calibration with variable metering volumes and constant concentration can be used.

The syringe pump is highly linear. Due to the large metering range of the syringe pump a wide calibration range can also be covered.

Multiple point calibration with constant sample volume

A multiple point calibration with **constant sample volume** and **variable concentrations** can also be carried out.

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

3.4.2 Day factor

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

The day factor is calculated in accordance with the equation (3).

$$F = \frac{c_{nominal}}{c_{actual}} \qquad (3)$$

3.4.3 Calibration method in multiWin

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

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

The calibration function is calculated in relation to the mass m per injected sample. Linear and quadratic calibration functions are calculated in accordance with the equations (4) and (5) through regression calculation.

$$c = (k_1 * I_{net} + k_0)/V \quad (4)$$

$$c = (k_2 * I_{net}^2 + k_1 * I_{net} + k_0)/V \quad (5)$$

c	target concentration of the standards
V	sample volumes
I_{net}	net integer
k_0, k_1, k_2	calibration coefficient

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

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

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

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

TC/NPOC

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

The calibration functions are calculated in accordance with the equations (4) or (5); the following applies:

$$c_{TC} = f(I_{TC}) \quad (6)$$

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

TIC

The TIC channel is calibrated. The calibration functions are calculated in accordance with the equations (4) or (5); the following applies:

$$c_{TIC} = f(I_{TIC}) \quad (7)$$

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

TOC (Diff)

Generally separate calibration functions are calculated for the channels TC and TIC in accordance with the equations (4) or (5). The equations (6) and (7) apply.

The calculation of the analysis results is based on the calculated calibration functions for TC and TIC. The TOC content is then the result of the equation (8).

$$c_{TOC} = c_{TC} - c_{TIC} \quad (8)$$

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

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

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

NPOC plus

The method NPOC plus is calibrated the same way as the method TOC(Diff). Since the TIC is largely purged when using the NPOC plus method, it is important to calibrate a low operating range for the TIC calibration.

- separate calibration of TIC and TC channel
- measurement of samples and calculation of the analysis results
  - Purging the acidified sample (3 to 5 min)
  - detection of the remaining TIC content – concentration is calculated in accordance with the calibration curve
  - detection of the remaining TC content – concentration is calculated in accordance with the calibration curve
  - calculation of the TOC content in accordance with the equation (8) from the calculated concentration difference

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

3.4.4 Method characteristics

Remaining standard deviation

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

Standard deviation of the method

The standard deviation of the method describes in a unique and general way the quality of the calibration. For the unique evaluation of the quality the standard deviation of the method must be used.

Method variation coefficient

The variation coefficient of the method (relative standard deviation of the method) should be used for the comparison of different calibrations with different calibration ranges.

Correlation coefficient

The correlation coefficient compares the dispersion of the calibration measuring points of the regression function with the total dispersion of the calibration. If all calibration measuring points are on the calculated regression function, then the correlation coefficient is +1 or -1. For positive correlation coefficients the regression function is increasing, for negative ones it is decreasing.

Coefficient of determination

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

Linearity

As proof of the linearity of the calculated regression function an adaptation test according to MANDEL is carried out in the control and analysis software multiWin. Here the reduction of the residual variance is checked using a quadratic regression.

Variance homogeneity

Variance homogeneity exists if the standard deviation is independent of the concentration, i.e. the variance is constant over the whole calibration range. In the control and analysis software multiWin the standard deviations of the calibration range (smallest and largest concentration or volume) are examined.

If linearity and variance homogeneity are present, a linear regression can be assumed. In this case it is possible to determine the verification, detection and determination limits of the calibration. In the control and analysis software multiWin the calculation rules of DIN 32645 (calibration rules) are used.

Limit of detection

The verification limit of the calibration specifies the lowest concentration that can be differentiated qualitatively from the zero point with a given probability. The verification limit should always be smaller than the lowest calibration measuring point.

Detection limit

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

Limit of quantitation

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

3.4.5 Other calculations

For all measurements where multiple injections are carried out the average value (AV), standard deviation (SD) and variation coefficient (VC) are calculated and displayed. For each sample a tenfold determination can be carried out as a maximum.

Outlier selection

The control and analysis software multiWin offers the option for an automatic outlier selection. In the method a maximum limit for a variation coefficient or also for a standard deviation can be entered.

The minimum number of measurements agreed in the method will be carried out. If the distribution of the measured values is then above the agreed value (SD or VC) additional injections are carried out from the same sample until the specified maximum number of measurements has been reached.

After each measurement the variation coefficient or standard deviation are calculated for all combinations of measurements. If the variation coefficient or the standard deviation of at least one combination is smaller than the specified maximum variation coefficient or standard deviation, no further measurements are carried out.

The combination of measurements with the smallest variation coefficient or the smallest standard deviation is used to calculate the analysis results. The unused measurements are considered as outliers and deleted.

Mean value

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

3.5 Blank Values

3.5.1 Blank water values

Preparation blank

Especially for measurements with low TOC concentrations (µg/l range), there is a significant TOC content in the water used for preparing the standard. The weighed in standard concentration and the TOC blank value of the preparation water are often in the same order of magnitude. This blank value can be taken into account during calibration.

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

$$I_{net} = I_{gross} - I_{PreparationWater} \quad (9)$$

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

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

Dilution blank

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

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

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

Use of the dilution blank

For every measurement the actual dilution water integer (I_{DBW}) is calculated from the dilution blank value in accordance with the sample volume and the dilution ratio used (equation (10)) and deducted from the experimentally determined raw integer (equation (11)). The raw integer determined for each measurement I_{raw} is corrected by the blank value of the dilution water used.

$$I_{DBV} = V_{DBV} * \left(V_{sample} - \frac{NumberUnitsPrimarySample}{NumberUnitsDilution} * V_{sample}\right) \quad (10)$$

$$I_{eff} = I_{raw} - I_{DBV} \quad (11)$$

V_{DBV} dilution blank value
V_{sample} sample volume
I_{eff} effective integer
I_{raw} raw integer
I_{DBV} dilution water integer

Definition of the dilution

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

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

Calculation of the sample concentration

To calculate the sample concentration c the sample volume and the dilution ratio are used (equation (12)).

$$c = \frac{m}{V_{sample}} * \frac{NumberUnitsDilution}{NumberUnitsPrimarySample} \quad (12)$$

For the linear calibration function (equation (4)) the result is then equation (13).

$$c = \frac{k_1 * I_{eff} + k_0}{V_{sample}} * \frac{NumberUnitsDilution}{NumberUnitsPrimarySample} \quad (13)$$

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

3.5.2 Reagent blank value

Especially for the measurement of low TOC concentrations the blank value (TIC/TC content) of the reagents used must not be neglected. These blank values can be taken into account for all measurements.

- H_3PO_4 (reagent for TIC branch) – IC blank value
- $Na_2S_2O_8$ (reagent for TC branch, i.e. UV reactor) – TC blank value

Measuring the reagent blank value is preferable to manual feeding. In the measured blank values the calculated blank values (area unit = FE) relate precisely to the metered volume of the corresponding reagent.

Reagent blank values are recalculated for every new preparation of the reagent(s), otherwise the value entered last will be used.

The reagent blank value can be calculated separately and entered in multiWin or automatically calculated prior to an analysis series. The consideration of the reagent blank value of the reagents used (H_3PO_4 and $Na_2S_2O_8$) can be individual or collective. For multiple measurements the sequential individual calculation is preferred. For the calculation of the IC value the blank value for H_3PO_4 (reagent for the TIC value) must be calculated. For the calculation of the TC value the blank value for $Na_2S_2O_8$ (reagent for the TC value, i.e. UV reactor) must be measured or entered.

3.5.3 Eluate blank value

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

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

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

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

If a sample measurement is carried out with a so-called eluate method, the integer of the blank value is deducted from the integer of the sample measurement (dependent on the injection volume) (equation (14)).

$$I_{eff} = I_{raw} - I_{EBV} \qquad (14)$$

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

3.6 System Suitability Test (SST)

System suitability tests are used to validate analytical methods and devices for documenting the suitability of the selected procedure.

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

The standards to be used and their concentrations are defined in the respective pharmacopeia, e.g. in the European Pharmacopeia or in the USP (United States Pharmacopeia). These define saccharose as an easily oxidizable and p-benzoquinone as a poorly oxidizable compound. The value returned for the recovery rate of the p-benzoquinone in relation to the value determined for saccharose must only be min. 85 % and max. 115 %.

Carry out the system suitability test as follows:

1. Prepare a reference solution from saccharose and TOC water containing 0.5 mg carbon per liter (corresponds to 1.19 mg saccharose in one liter of water).
2. Prepare a reference solution from b-benzoquinone and TOC water to examine the system suitability containing 0.5 mg carbon per liter (corresponds to 0.75 mg p-benzoquinone in one liter of water).
3. Determine the TOC concentrations of the reference solution r_s and the TOC water r_w in the selected mode (direct or differential method).

The percentage effectiveness of the system is calculated using the following formula:

$$E = \frac{r_{ss} - r_w}{r_s - r_w} \times 100 \qquad (15)$$

E... System effectiveness in %
r_s... TOC of the reference solution (saccharose)
r_{ss} TOC of the system suitability solution (p-benzoquinone)
r_w TOC the TOC water used (preparation water)

The system is suitable if the value derived from the formula above is > 85 % and < 115 %.

4 First commissioning

4.1 Site requirements

4.1.1 Installation conditions

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

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

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

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

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

4.1.2 Space requirement

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

The AS vario autosampler and the EPA sampler must be positioned on the right side of the analyzer. The AS 21 and AS 10 samplers are attached to the right-hand side of the analyzer. The layout of the other components can be adapted to the local conditions.

4.1.3 Energy supply

WARNING

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

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

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

4.1.4 Gas supply

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

The connection hose with outer diameter 6 mm and inner diameter 4 mm is included with the delivery.

4.2 Unpacking and placing the analyzer

CAUTION

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

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

ATTENTION

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

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

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

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

5 Connecting add-on devices

CAUTION

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

5.1 AS vario autosampler

WARNING

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

CAUTION

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

ATTENTION

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

Five different sample trays are available for the AS vario autosampler. A matching cannula holder is available for each sample tray.

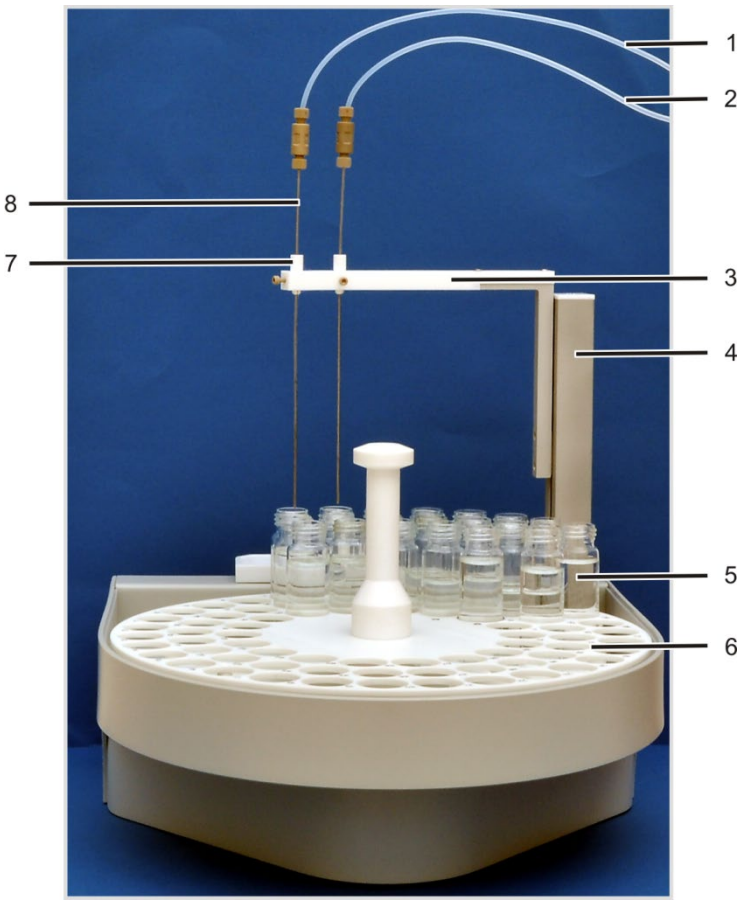
Sample tray

Max. no. of samples	20	52	72	100	146
Sample tubes	100 ml	100 ml	40 and 50 ml	20 ml	12 ml

Technical data

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

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



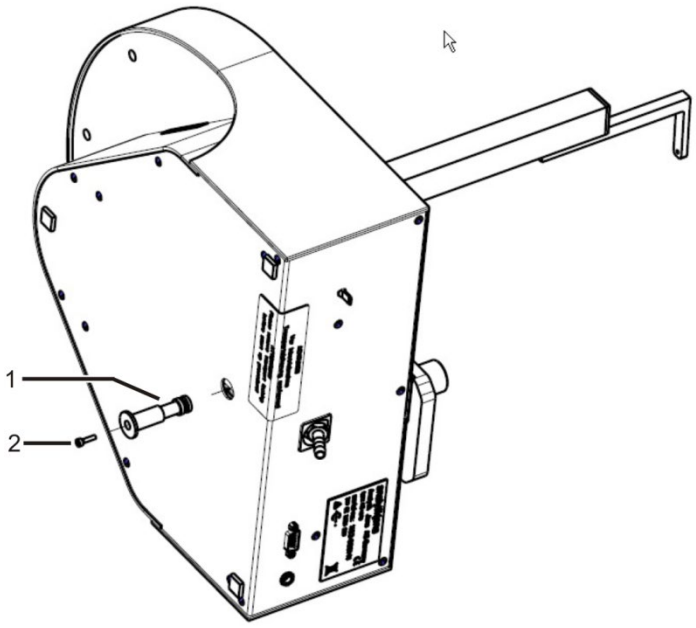
1 Connection hose to the analyzer (purging hose for NPOC measurements)
2 Connection hose to the analyzer (sample aspiration hose)
3 Cannula holder
4 Autosampler arm
5 Sample tubes
6 Sample tray
7 Sleeve
8 Cannula

Fig. 15 Layout of the AS vario autosampler

Removing the transport lock

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

1. Place the autosampler on the side as shown in Fig. 16.
2. Unscrew the screw (2 in Fig. 16) with the Allen wrench (included in the scope of delivery) and remove the red transport lock.
3. For commissioning replace the autosampler on the baseplate.



1 Transport lock
2 Screw M3x12

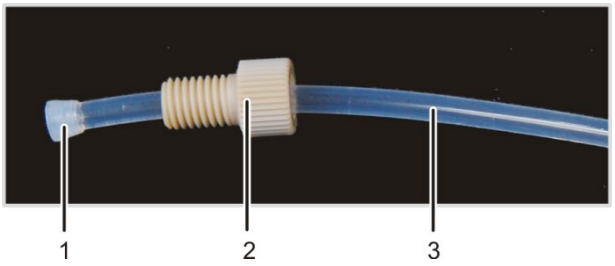
Fig. 16 Transport lock on autosampler AS vario

Commissioning the autosampler

4. Switch off the analyzer!
5. Plug cable on the low voltage side of the table power supply included in the delivery into the connection on the rear of the autosampler. Do not yet connect the power supply to the grid.
6. Plug the grounding conductor into the connection on the rear of the analyzer.
7. Connect the autosampler to the analyzer with the interface cable (port on the rear of the autosampler and "sampler" port on the rear of the analyzer).
8. Attach the outlet tube to the outlet connector on the rear of the autosampler. Insert the other end of the tube into the opening in the cover of the waste bottle. Attention: Position the outlet tube at a constant incline. If necessary shorten the tube. Tube must not dip in the liquid.
9. Place the sample tray onto the autosampler housing. Make sure it clicks into place.
10. Check that the correct cannula holder has been installed at the autosampler arm. To do so, compare the number engraved on the bottom with the max. number of sample tubes. They have to be identical.
11. Insert the cannulas with the matching sleeve into the cannula holder.
    - For NPOC measurements with parallel purging: Insert 1 cannula with sleeve into each of the two cannula holder positions (see Fig. 15)
    - For NPOC measurements with non-parallel purging: Insert both cannulas in one sleeve with two holes in the position on the right (see Fig. 17)

Fig. 17 Sleeve with 2 cannulas

12. Adjust the cannulas so that approx. one third of the cannulas is visible above the sleeve. Secure the cannulas by slightly tightening the screw.
13. Connect the two connection hoses to the analyzer to the cannulas:
    Hose no. 6 = sample aspiration hose
    Hose no. 10 = purging hose for NPOC measurements
    - Release the upper Fingertight connection of the cannula.
    - Guide the hose through the banjo bolt.
    - Slide the conical nipple with the conical side towards the banjo bolt onto the hose. The conical nipple and hose must be flush.
    - Retighten the Fingertight connection.



1 Conical nipple
2 Banjo bolt
3 Hose

Fig. 18 Hose in Fingertight connection

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

Before the first start the sampler must be adjusted (see "Adjusting the AS vario autosampler" section, p. 69).

5.2 Sampler AS 21

WARNING

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

CAUTION

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

ATTENTION

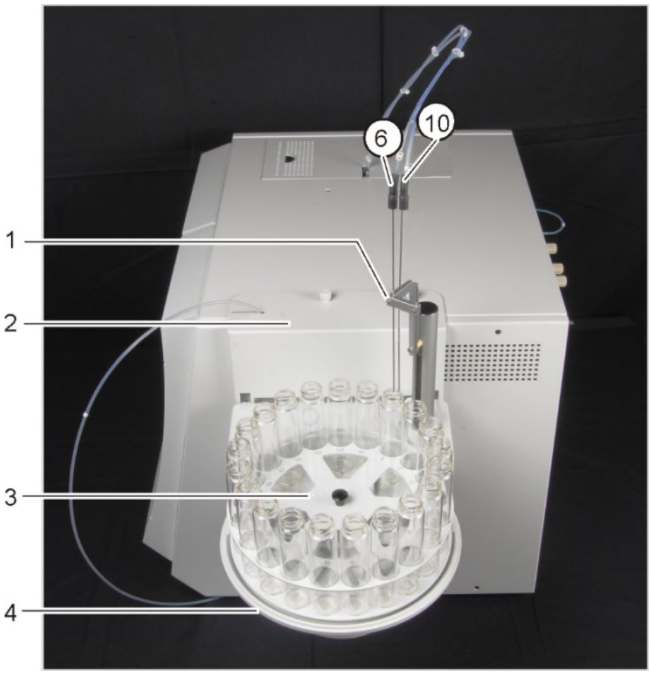
Do not obstruct the sampler during running operation. The drives might be damaged.

Technical data

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

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

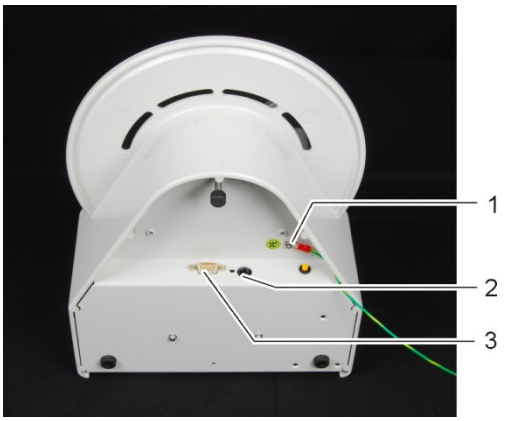
Layout



1 sampler arm with cannula holder
2 holder at the analyzer
3 sample tray
4 Drive unit

hose no. 6
sample intake hose
hose no. 10
purging cannula

Fig. 19 Layout of the sampler AS 21



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

Fig. 20 Connections at the bottom of the sampler AS 21

Installation at the analyzer

1. Screw the holder to the right-hand side of the analyzer.
2. Place drive onto the holder.
3. Plug the grounding conductor into the connection on the rear of the analyzer (8 in Fig. 13 p. 23).
4. Plug cable on the low voltage side of the table power supply included in the delivery into the connection at the bottom of the sampler. Do not yet connect the power supply to the mains.
5. Plug the interface cable into the bottom of the equipment. Connect the sampler to the interface at the rear of the analyzer (11 in Fig. 13 p. 23).
6. Place a sample tube into position 1 of the sample tray.
7. Insert the cannulas into the autosampler arm according to Fig. 19.
8. Manually adjust the height of the cannulas so that the cannula tips protrude 1 – 2 cm over the tube edge and cannot touch the tubes when the autosampler arm is moving. Secure the cannulas by slightly tightening the screw.

   Attention: the screws must not bend the cannulas under any circumstances.
9. Connect the switching power supply to the mains. Switch on the switching power supply.
10. Check the configuration via the menu command INSTRUMENT ▸ SYSTEM INFORMATION in the window SET-UP INFO. If necessary, modify the configuration:
    - Exit the program multiWin.
    - On the Windows user interface start the SET-UP TOOL under START ▸ PROGRAM FILES ▸ MULTIWIN ▸ MULTIWIN SET-UP TOOL.
    - In the list SAMPLER select the sampler type.
    - Exit the SET-UP TOOL with [CREATE].
    - Start the multiWin program and select the CONFIGURATION ▸ EDIT OPTIONS menu command to open the OPTIONS window on the ANALYZER COMPONENTS tab. In the SAMPLER group, select the correct tray and tube size. Exit the OPTIONS window with [OK].
11. Adjust the immersion depth of the cannulas in the tube (z direction) in multiWin.
    - Select the INSTRUMENT ▸ SAMPLER ALIGNMENT menu command to open the window with the same name.

- In the PLEASE SELECT POSITION NEEDING ADJUSTMENT GROUP, select POSITION 1.
- Click [POSITION 1 ADJUST].
  The autosampler arm now lowers the cannulas into the tube in position 1.
- If necessary, increase or decrease the z values. Click [POSITION 1 ADJUST] again after each change to verify the change.
- Once adjustment is complete, click [SAVE] to close the window.

Retrofit to the function "parallel purging"

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

Fig. 21 Cannula holder for parallel purging

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

1. Screw the cannula holder (7) to the autosampler arm if it is not preinstalled.
2. Push the spacer (4) onto the two cannulas. Lightly secure the spacer below the hose connections with the fixing screw, so that it cannot move.
3. Insert the cannulas into the cannula holder according to the figure and attach them only lightly using the knurled head screws.
4. Place two sample cups into positions 1 and 2 of the sample tray under the two cannulas.
5. Manually adjust the height of the cannulas so that the cannula tips protrude 1 – 2 cm over the tube edge and cannot touch the tubes when the autosampler arm is moving.
6. Adjust the immersion depth of the cannulas in the tube (z direction) in multiWin.
   - Select the INSTRUMENT ▸ SAMPLER ALIGNMENT menu command to open the window with the same name.
   - In the PLEASE SELECT POSITION NEEDING ADJUSTMENT GROUP, select POSITION 1.

- Click [POSITION 1 ADJUST].
  The autosampler arm now lowers the cannulas into the tube in position 1.
- If necessary, increase or decrease the z values. Click [POSITION 1 ADJUST] again after each change to verify the change.
- Once adjustment is complete, click [SAVE] to close the window.

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

   Attention: The hoses may not hinder the movement of the autosampler arm.

5.3 Sampler AS 10

WARNING

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

CAUTION

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

ATTENTION

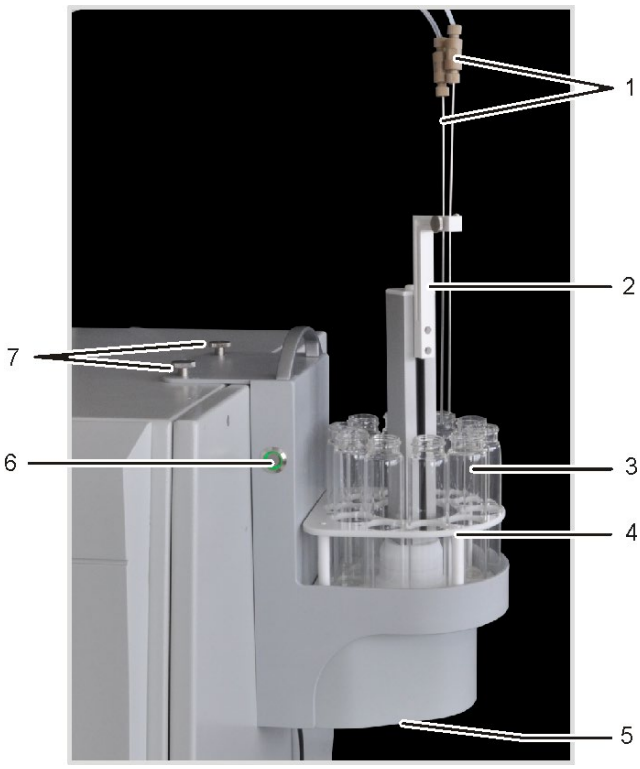
Do not obstruct the sampler during running operation. The drives might be damaged.

Technical data

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

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

Layout



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

Fig. 22 Layout of the AS 10 autosampler

Installation at the analyzer

1. Plug cable on the low voltage side of the table power supply included in the delivery into the connection at the bottom of the autosampler and connect the power supply to the grid.
   Make sure the AS 10 is switched off. (The green LED of the On/Off must be off.)
   Connect the autosampler to the analyzer with the interface cable (port on the bottom of the autosampler and "sampler" port on the rear of the analyzer).
2. Screw the sampler with the two fastening screws to the right-hand side of the equipment.
3. Place a sample tube into position 1 of the sample tray.
4. Insert the cannulas into the autosampler arm. Manually adjust the height of the cannulas so that the cannula tips protrude 1 – 2 cm over the tube edge and cannot touch the tubes when the autosampler arm is moving.
5. Secure the cannulas by slightly tightening the screw.
6. Switch on the AS 10 at the On/Off switch.
7. Check the configuration in the multiWin program via the INSTRUMENT ▸ SYSTEM INFORMATION menu command in the SET-UP INFO window. If necessary, modify the configuration:
   - Exit multiWin.
   - In the Windows user interface, start the SET-UP TOOL under START ▸ PROGRAM FILES ▸ MULTIWIN ▸ MULTIWIN SET-UP TOOL.
   - Select the sampler type in the SAMPLER list.
   - Exit the SET-UP TOOL with [CREATE].
   - Start the multiWin program and select the CONFIGURATION ▸ EDIT OPTIONS menu command to open the OPTIONS window on the ANALYZER COMPONENTS tab. In the

SAMPLER group, select the correct tray and tube size. Exit the OPTIONS window with [OK].

8. Adjust the immersion depth of the cannulas in the tube (z direction) in multiWin.
   - Select the INSTRUMENT ▸ SAMPLER ALIGNMENT menu command to open the window with the same name.
   - In the PLEASE SELECT POSITION NEEDING ADJUSTMENT group, select POSITION 1.
   - Click [POSITION 1 ADJUST].
     The autosampler arm now lowers the cannulas into the tube in position 1.
   - If necessary, increase or decrease the z values. Click [POSITION 1 ADJUST] again after each change to verify the change.
   - Once adjustment is complete, click [SAVE] to close the window.

5.4 EPA sampler

WARNING

Always disconnect the power plug before opening the device!

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

CAUTION

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

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

ATTENTION

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

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

Technical data

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

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

Layout

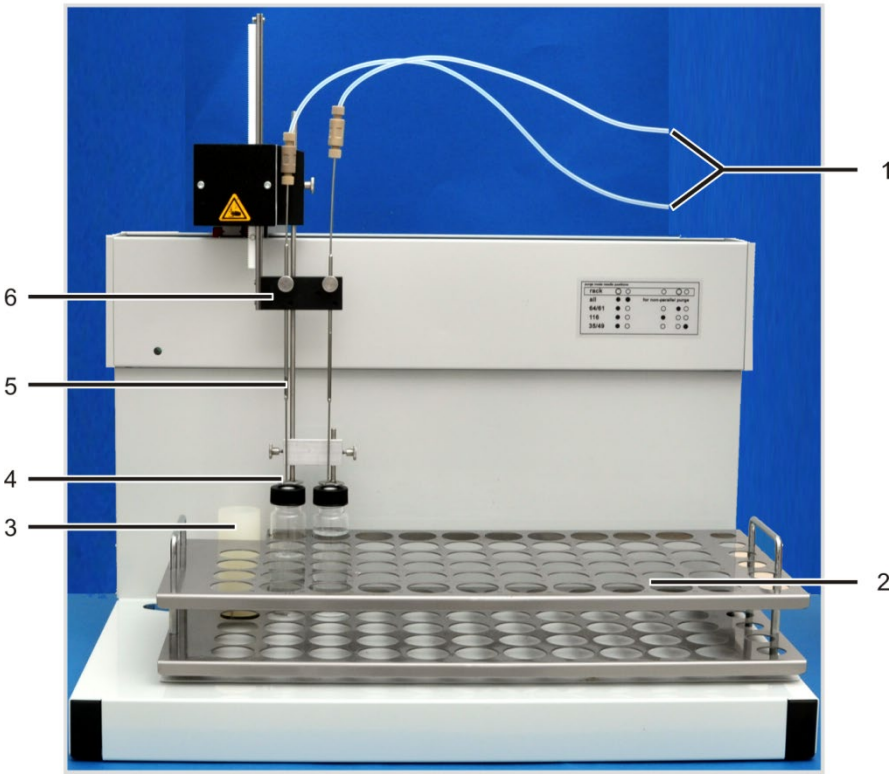
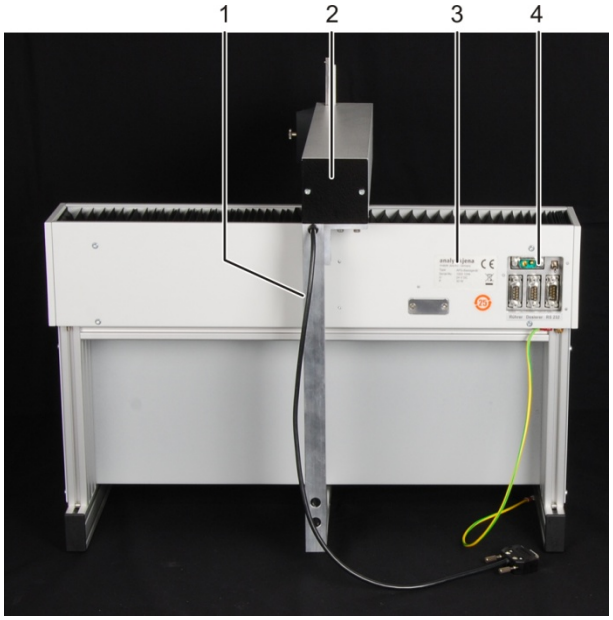


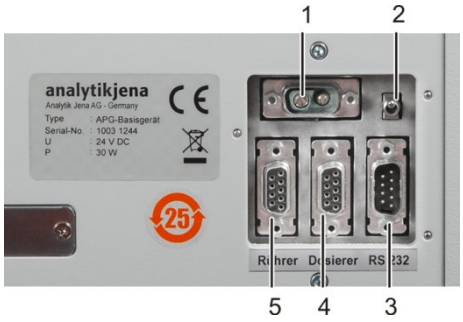
Fig. 23 EPA sampler

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



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

Fig. 24 Rear of the EPA sampler

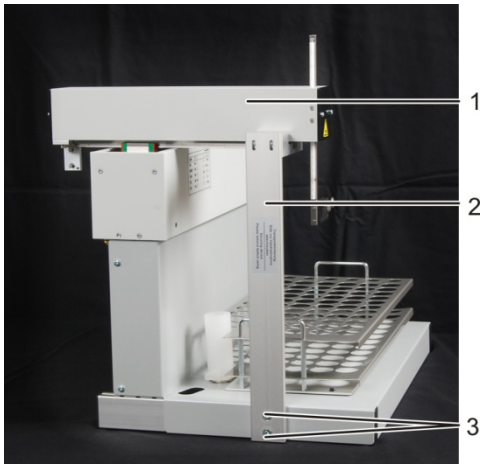


1 Connection to power supply unit
2 Equipment switch
3 Connection to the analyzer
4 Not used
5 Stirrer connection

Fig. 25 Electrical connections on the rear of the EPA sampler

Setting up the EPA sampler

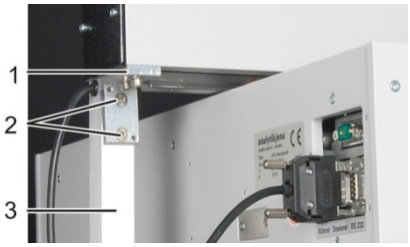
1. Remove the transport lock!
   - Remove the two countersunk screws with the A/F3 hexagon head wrench supplied.
   - Remove the complete transport retaining clip and retain the transport lock well (for transport in case of a service requirement etc.).



1 Autosampler arm
2 Transport retaining clip
3 Screws

Fig. 26 Transport lock

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

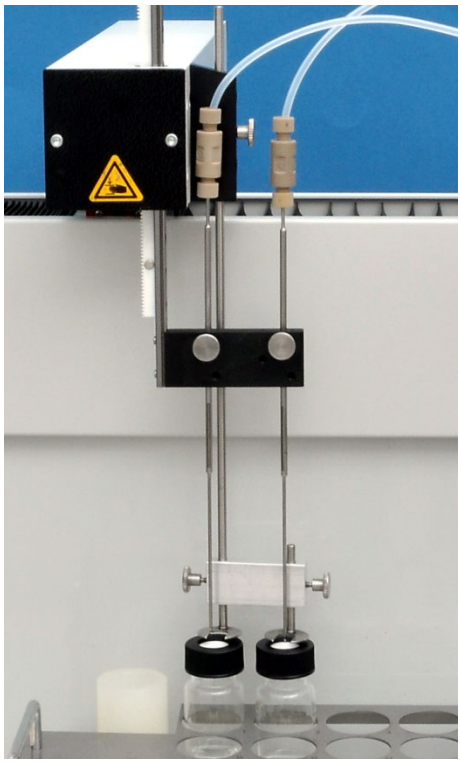


1 Bracket at the autosampler arm
2 Countersunk screws
3 Stirring arm

Fig. 27 Fitting the stirring arm to the autosampler

3. Place the sampler to the right of the analyzer.

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

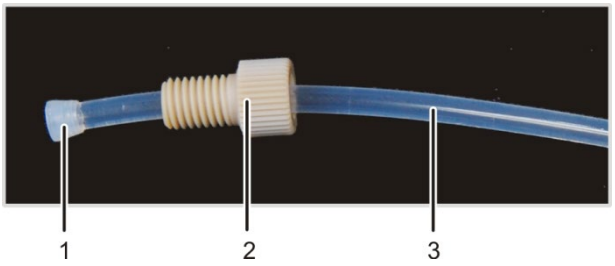


For NPOC measurements with parallel purging:
Insert 1 cannula into each of the two positions in the cannula holder

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

11. Clamp the two cannulas high enough in the holder to prevent them dipping into the tubes (basic position).
12. Connect the two connection hoses to the analyzer to the cannulas:
    Hose no. 6 = sample aspiration hose
    Hose no. 10 = purging hose for NPOC measurements
    - Release the upper Fingertight connection of the cannula.
    - Guide the hose through the banjo bolt.
    - Slide the conical nipple with the conical side towards the banjo bolt onto the hose. The conical nipple and hose must be flush.

- Retighten the Fingertight connection.



1 Conical nipple
2 Banjo bolt
3 Hose

Fig. 28 Hose in Fingertight connection

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

Before the first start the sampler must be adjusted (see "Adjusting the EPA sampler" section, p. 72).

6 Operation

6.1 General information for the analysis work

Observe the following during analysis:

- When analyzing samples with high acidic or saline content, aerosol can be formed in the TIC condensation vessel. The capacity of the halogen trap is depleted within a relatively short time. The water trap also clogs up quickly. Both components have to be renewed frequently if this is the case. Where possible such samples should be diluted prior to the measurement (e. g. 1:10).
- To acidify the samples, only sulfuric acid (H_2SO_4) p. A. c = 2 mol/l, prepared from H_2SO_4 p. A. (conc.) and TOC water may be used.
- For the TIC detection only 10 % ortho-phosphoric acid (H_3PO_4), made from ortho-phosphoric acid (concentrated) p. A. and TOC water, must be used.
- Only clean, particle-free glass containers (volumetric flasks, vials) are to be used for the creation and storage of substances.
- When preparing and storing solutions in the range of < 1 mg/l it must be noted in particular that the concentrations of the solutions may be slightly modified by components of the laboratory air (CO_2, organic vapors).
  You can take the following precautions against this:
  - Keep the head room above the liquids as small as possible.
  - Cover the sample vials on the sample tray with foil during autosampler operation (for example aluminum foil).
  - In manual mode aerate the head room of the sample:
    Connect the aeration hose to the "pump" connection (5 in Fig. 13 p. 23) and enter the other end of the aeration hose into the head room of the sample.

  When aerating the head room do not submerge the aeration hose into the liquid.
  - Remove the source of organic vapors.

6.1.1 Reagents and materials required

The operator is responsible that everything not included directly in the scope of delivery yet necessary for the operation of the analyzer will be available during the installation of the device. This applies in particular to the first supply of consumables.

ATTENTION

The analyzer will be damaged if different reagents are used. Only the specified reagents must be used!

The operator must provide the following reagents and materials:

- only chemicals of the degree of purity "for analysis" may be used
- only clean, particle-free glass containers (volumetric flasks, vials) may be used for the preparation and storage of the solutions.

- TOC water (ultrapure water) as system water (device-internal rinsing water) and for the preparation of standards with the following requirements (conductivity < 1.0 µS/cm (at 25 °C), TOC < 0.1 mg/l
- orthophosphoric acid (H_3PO_4) 10 % prepared from orthophosphoric acid (concentrated) p. A. and TOC water
- sodium peroxodisulfate solution $Na_2S_2O_8$ (concentration 80 g/l) and 10 ml sulfuric acid H_2SO_4 (concentration 2 mol/l) as reagent for UV digestion
- sulfuric acid H_2SO_4 (concentration 2 mol/l) to acidify the sample
- standard substances (e. g. potassium hydrogen phthalate, sodium carbonate/sodium hydrogen carbonate, saccharose)
- gas supply (nitrogen/argon)
- acid-proof safety gloves, safety goggles
- PC, monitor and printer for multi N/C pharma UV (if delivered without PC)

6.2 Switching on the analyzer (standard commissioning)

ATTENTION

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

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

Always check the following before switching on the analyzer:

- The waste hose is connected to a suitable waste container or drain, clear drainage is ensured and the capacity of the waste container is adequate.
- The gas supply is connected in accordance with regulations and the preliminary pressure is 4 to 6 bar.
- The reagent bottles contain sufficient reagents for the analysis:
  - 1 ml phosphoric acid (H_3PO_4) for the TIC detection per analysis
  - 2 ml sodium peroxodisulfate ($Na_2S_2O_8$) for TOC, TC, NPOC detection
- The halogen trap is connected, filled with copper and brass wool and still usable (see safety note above).
- The hoses in the analyzer are connected properly and in good working order, e.g.:
  - ultrapure water: hose no. 5
  - reagent bottle with H_3PO_4: hoses no. 2 and A
  - reagent bottle with $Na_2S_2O_8$: hoses no. 3 and B
  - sample intake cannula: hose no. 6
  - sample purge cannula: hose no. 10

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

Position a sample within reach and switch the analyzer on as follows:

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

   In the window SYSTEM STATE the displays of the components which are not yet ready are shown in read during initialization. During the start-up phase of the analyzer the external communication with the program is blocked. The NDIR detector requires a start-up period of approx. 10 minutes.
7. If the analyzer is not ready for measurements after 15 minutes (one or several components are still shown in red in the window SYSTEM STATE), check the hose connections and carry out a fault finding exercise in accordance with the notes in section "Fault removal" p. 86.
8. If necessary, adjust the NPOC purge flow (see section "Adjust the NPOC purge flow" p. 71).

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

6.3 Switching off the analyzer

6.3.1 Switching to standby mode

ATTENTION

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

Frequently switching the UV lamp on and off during brief intervals between measurements reduces the service life of the UV lamp.

ATTENTION

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

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

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

   The PROGRAM END window opens.
2. Purging the analyzer:

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

3. Enable the option STAND-BY ANALYZER and confirm using [OK].
   ✓ The analyzer remains in standby mode.

The sample intake hose is purged with ultrapure water. The rest of the system remains filled. The UV lamp is switched off and the gas flow is reduced.

6.3.2 Switching off before longer periods of rest

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

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

   The PROGRAM END window opens.

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

   The sample intake hose is purged with ultrapure water. The UV reactor and the TIC condensate container are drained. The UV light and the gas flow are turned off. When activating the option REVERSE RINSE ANALYZER the syringe pump is flushed as well. These processes take about 2-3 minutes.

4. Switch off the analyzer using the power switch.
   ✓ The analyzer is switched off completely.

6.4 Carrying out the calibration

6.4.1 Preparing and starting the calibration

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

Carry out the calibration as follows:

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

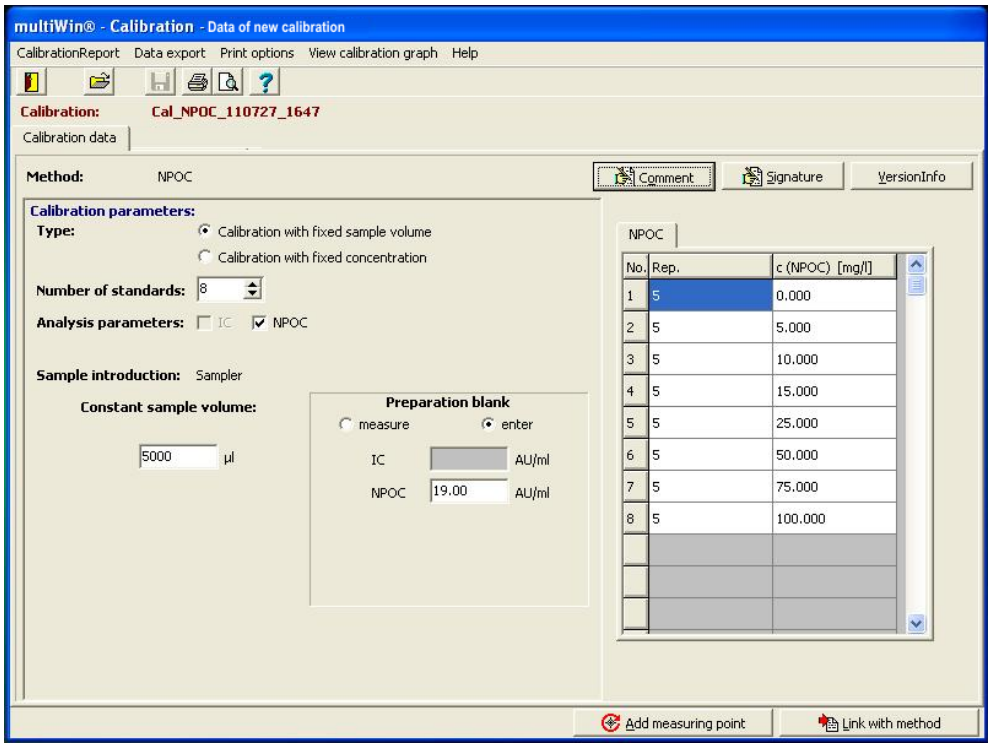


Fig. 29 Calibration window - DATA OF NEW CALIBRATION

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

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

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

5. In the input field NUMBER OF STANDARDS enter the number of calibration points.
6. Select the ANALYSIS PARAMETERS of the loaded method to be calibrated.

   Not all parameters need necessarily be calibrated.
7. Under SAMPLE INTRODUCTION the type of sample supply is indicated. The indication is for information only and must not be modified.
8. In the group PREPARATION BLANK select how the preparation water blank value of the standard should be taken into account.

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

   The preparation water blank value must be specified standardized to 1 ml. If the preparation water blank value is not taken into account enter a 0 in the input field.
9. Complete the calibration table for each parameter to be calibrated in accordance with the standard solutions provided.

   For calibrations with fixed concentrations and different standard volumes the **smallest volume must be at least 1.6 ml!**

   In the column REP. the number of repeat measurements configured in the method is entered automatically. If the outlier selection is enabled in the method, the maximum number is entered. The number of repeat measurements can be manually changed individually for each standard.
10. If necessary, save your calibration table with the menu commands CALIBRATIONTABLE ▸ SAVE CALIBRATIONTABLE or CALIBRATIONTABLE ▸ SAVE CALIBRATIONTABLE AS....

    Calibration tables are automatically given the extension *.kaltab and are saved under ...\CALIBRATION\TABLES.
11. Click on the button [MEASUREMENT] and then follow the instructions on the screen.

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

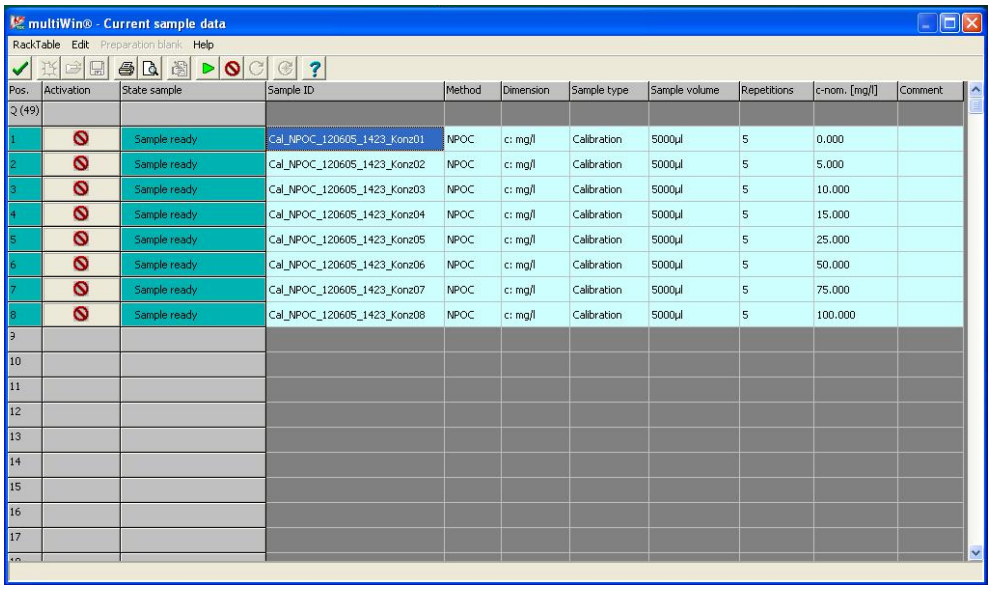


Fig. 30 Window CURRENT SAMPLE DATA (with sampler operation)

12. Release the calibration standards in the window CURRENT SAMPLE DATA with  and exit the window with the button  .
13. After opening the window MEASUREMENT click on the button [START F2].
    - ✓ The calibration process starts.

6.4.2 Displaying calibration results

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

The window CALIBRATION – CALIBRATION SETTINGS has the tab CALIBRATION DATA with the configurations for the calibration and the tab CALIBRATION RESULT with the compilation for each calibrated parameter.

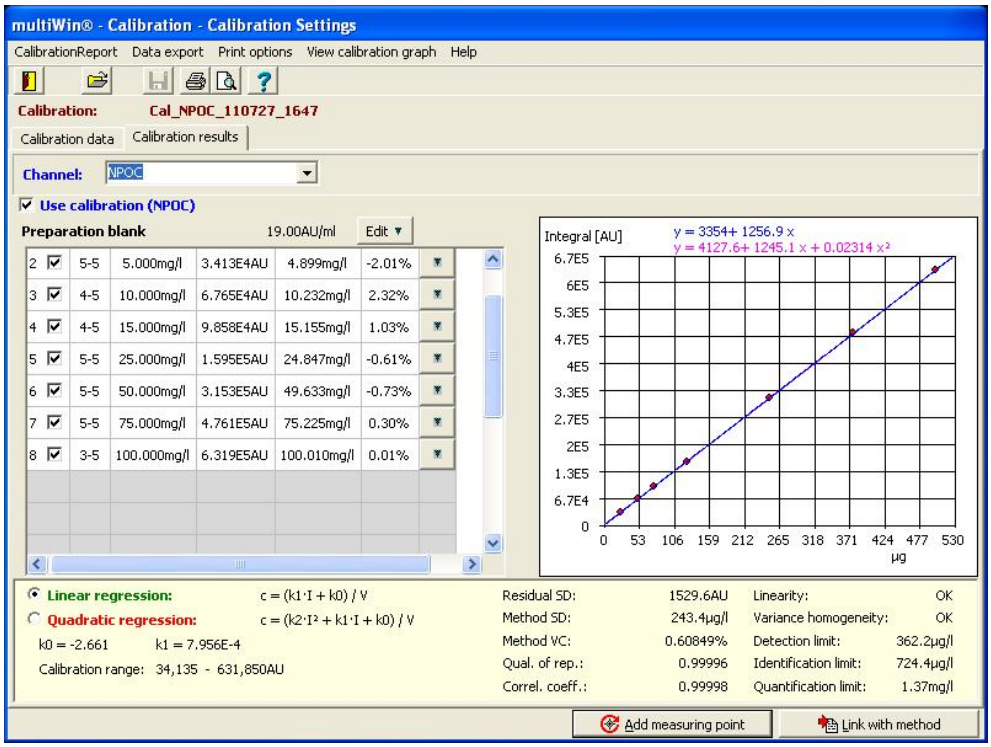


Fig. 31 CALIBRATION window - Data of the completed calibration

Tab Calibration results

Result table	The following are displayed: number of detections target concentration used for constant sample volume or sample volume used for constant concentration average value of the area integers average values of the calculated concentrations percentage deviation of the calculated concentration from the target concentration
Linear regression/ quadratic regression	Dependent on the methodology selected the regression calculation and determination of the method characteristics are based on individual values or average values of the net integer. For the selected regression type the respective calibration coefficients are displayed.
Calibration diagram	The regression graph can be displayed in accordance with the regression for the program-internal calibration coefficient determination (x axis integer; y axis mass) or in accordance with the determination of the method characteristics (x axis mass; y axis integer). The change-over of the view takes place in the menu View calibration graph.
Method characteristics	■ Linearity test The linearity test is carried out if at least four calibration measurement points are used for the analysis. An adjustment test according to MANDEL is carried out with a significance level of P = 99 %. The result of the linearity test (OK = correct, FALSE = incorrect) serves as a recommendation for the selection of the regression type; the recommended regression is shown in green. ■ Variance homogeneity The test for the variance homogeneity of the calibration is only carried out when determining the method characteristics from

average values. It is also necessary to use at least two individual detections for the analysis for the selected calibration measuring points with the smallest and largest target concentration.
The test is carried out at a significance level of P = 99 %.

- Verification/detection and determination limit
  In multiWin the calculation rules of DIN 32645 (calibration function) are used with a significance level of P = 95 %.
  For the calculation of the determination limit a relative result uncertainty of 33.3 % is being assumed (factor k = 3). Other method characteristics see also section "Method characteristics" p.30.

6.4.3 Editing an existing calibration

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

The following items can be edited during a calibration:

- Selection of the regression type

  Either linear or quadratic regression can be selected (see Fig. 31). For the selected regression type the respective calibration coefficients and method characteristics are displayed.

- Disabling individual measuring points

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

- Disabling individual measured values

  By clicking the button at the end of each line of the results table you can view the individual measured values (see Fig. 32). Individual values can be disabled by removing the (✓) in the column **use**.

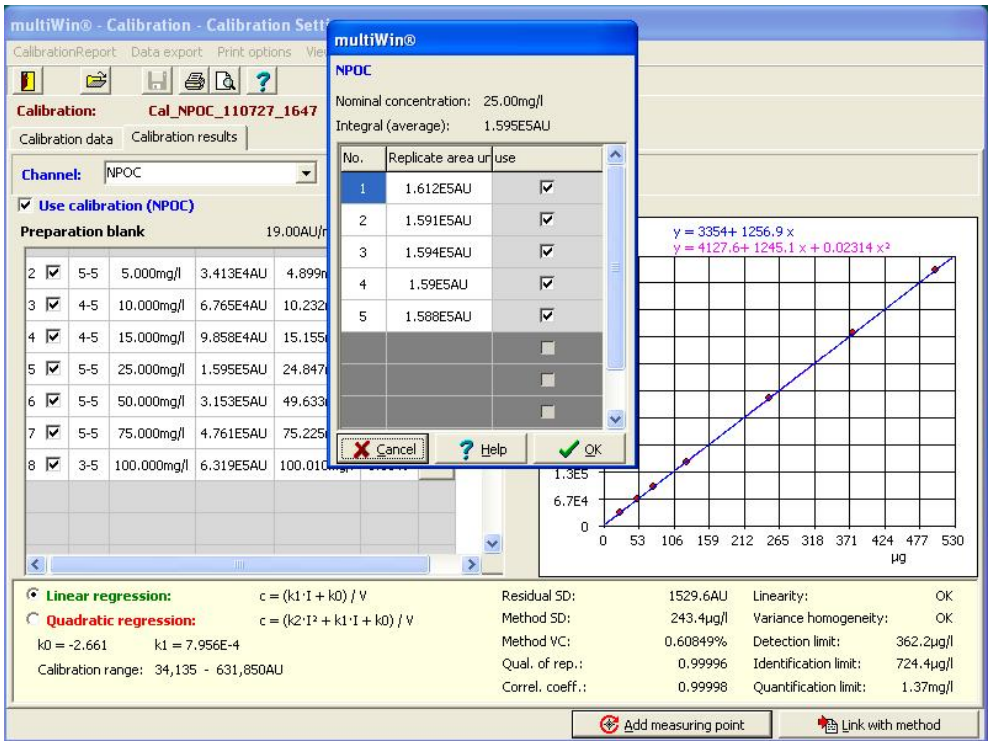


Fig. 32 **Disabling individual measured values of a calibration**

- Enabling/disabling measured values for preparation water

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

- Adding measuring points

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

  Measuring points can only be added individually.

6.4.4 Transferring calibration parameters to a method

Transfer calibration parameters to a method as follows:

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

   Up to three calibration ranges for each parameter can be stored in a method. It must be noted that the ranges should overlap and have no gaps! Every calibration range must be transferred individually, i.e. select calibration range, link, and transfer.
   Using calibration function with quadratic regression only one calibration range can be stored in a method.

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

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

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

4. Answer the subsequent query "Link to calibrated method?"
   - [YES] the link is made with the calibrated method (default)
   - [NO] the calibration parameters are linked to a method individually selected

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

5. In the window LINK WITH METHOD*:* that opens the existing current calibration coefficients (right-hand column) and the newly determined calibration coefficients (left-hand column) are displayed and can be compared (Fig. 33).

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

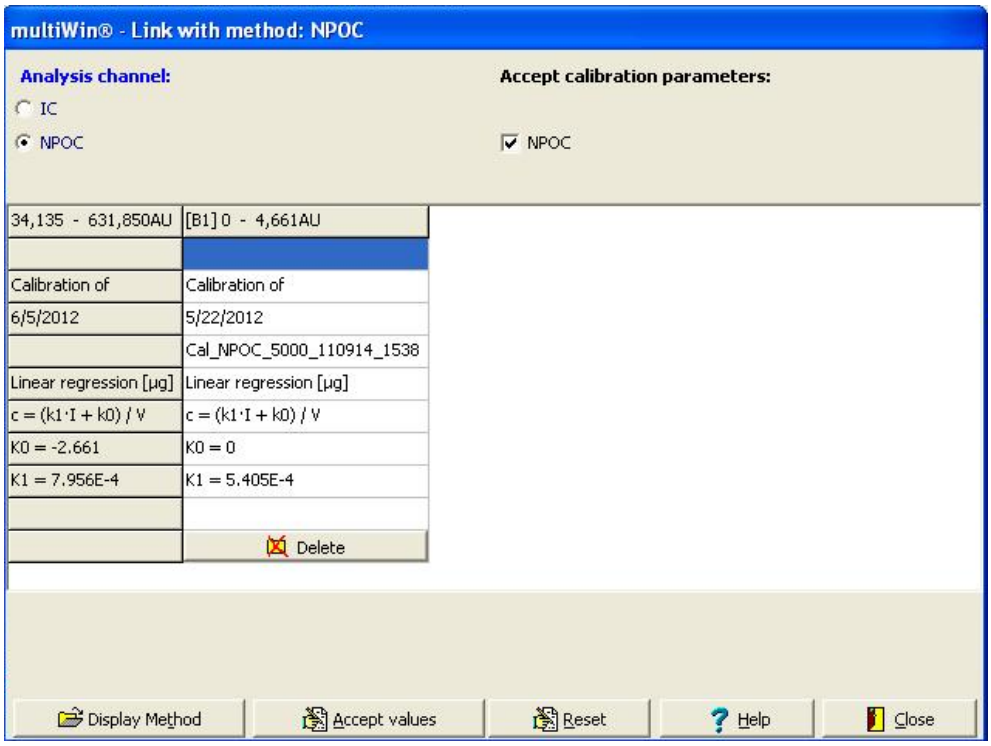


Fig. 33 Window Link to Method

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

No calibration range exists	Transfer the currently determined calibration data with the button [ACCEPT VALUES]. The same calibration coefficients appear in the left-hand and right-hand columns.
One or two calibration ranges exist	Extend the existing calibration range: Amend the new calibration coefficients with the button [Accept values]. Irrespective of the areas the software integrates the new range into the existing ones. Check on the basis of the calibration ranges that a seamless linking of several ranges has taken place. Replace existing calibration range: Delete the calibration range using the button [DELETE]. Then proceed as in "Extend existing calibration range".

Three calibration ranges exist	A maximum of three calibration ranges can be stored for each parameter in a method. In this case the ranges can only be replaced. Delete the range to be replaced in the right-hand columns using the button [DELETE]. Transfer the currently determined calibration data with the button [ACCEPT VALUES]. Check on the basis of the calibration ranges that a seamless linking of several ranges has taken place.

The following generally applies:

- Pressing the button [ACCEPT VALUES] causes an automatic allocation of the calibration ranges by the software.
- By pressing the button [DELETE] you first make an initial selection which range should be replaced.
- A SEAMLESS LINKING means that the top end of the area of one calibration range corresponds to the bottom end of the area of the next calibration range (see Fig. 34 table, first line)
- The accepted calibration parameters are used for the calculation of all subsequent analyses with this method.

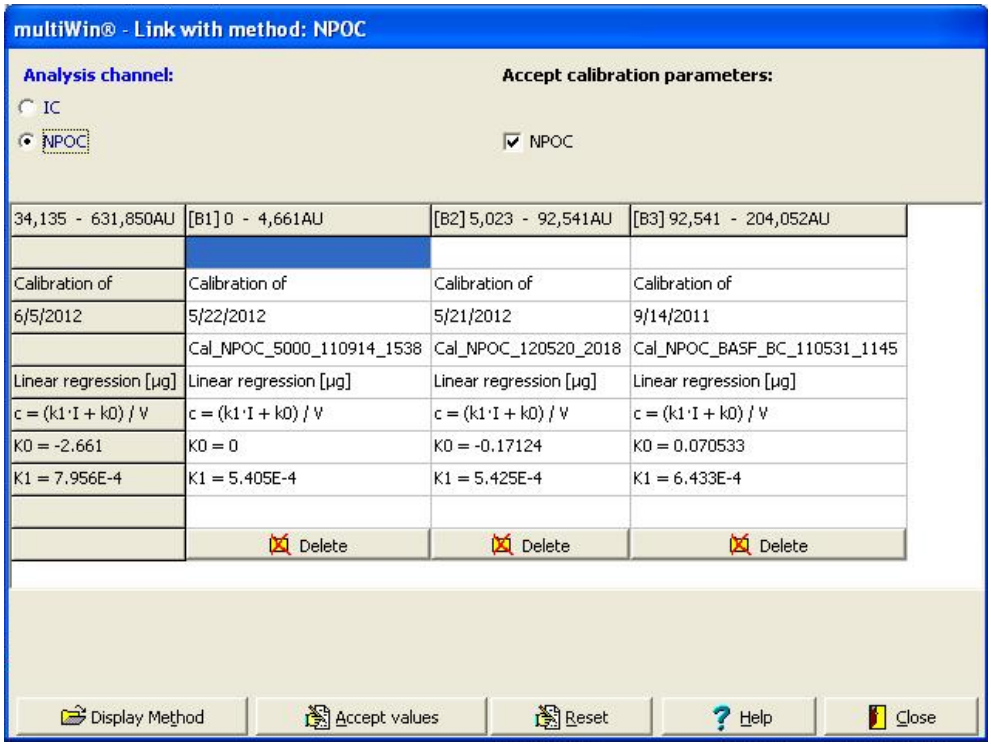


Fig. 34 Window LINK WITH METHOD with three areas

6.4.5 Managing calibration data

Printing calibration data

Print the calibration report as follows:

1. In the window CALIBRATION – CALIBRATION SETTINGS enable the option USE CALIBRATION.
2. Define the scope of printing under the menu PRINT OPTIONS:
   - Print calibration graph and/or
   - print individual integers for each calibrated channel
3. Start the printout with the menu command CALIBRATIONREPORT ▸ PRINT.

Exporting calibration files

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

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

Reopening a calibration report

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

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

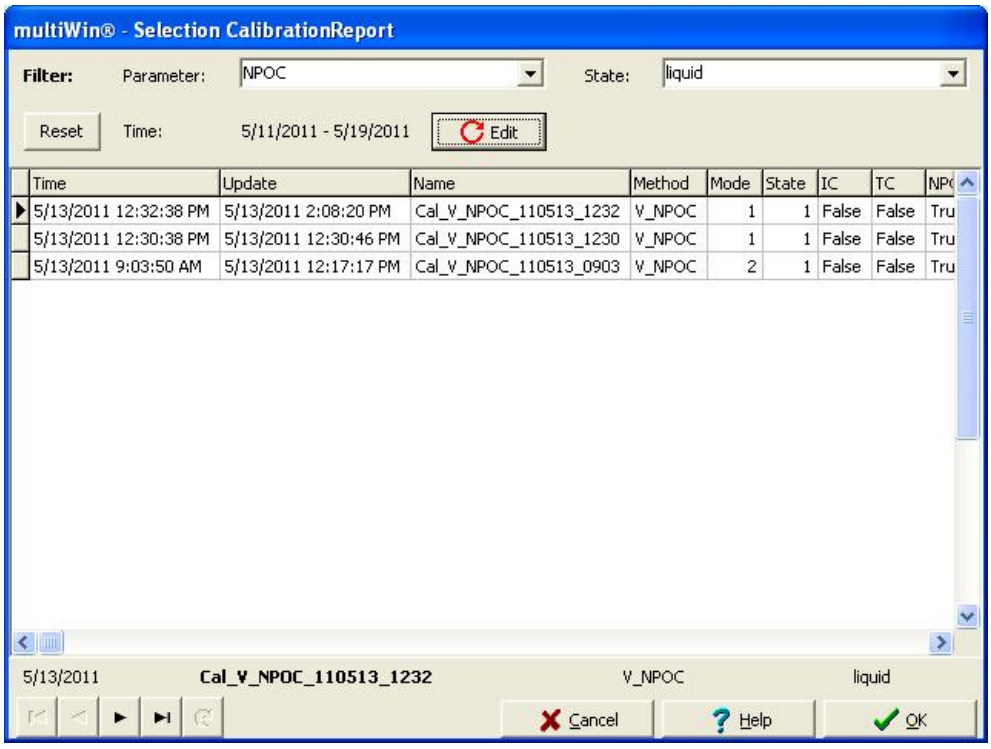


Fig. 35 Window CALIBRATION REPORT SELECTION

3. Highlight the corresponding calibration report and click on the button [OK].
   ✓ The calibration report is displayed.

6.5 Performing measurements

6.5.1 Measurement with manual sample supply

Carry out a measurement with manual sample supply as follows:

1. Insert the sample intake cannula and the purging cannula into the sample.
2. Using the menu command METHOD ▸ NEW create a new method or load an existing method.
   To do so open via the menu command METHOD ▸ LOAD the database window METHOD SELECTION, highlight the desired method and confirm the selection by clicking the button [OK].
3. In the window SYSTEM STATE select the manual sample supply by clicking the button [MANUAL].
   ✓ This is followed by the initialization of the analyzer.
4. In the window SYSTEM STATE check the following entries:
   - Visual bank - OK
   - Gas flow - OK
   - UV lamp - ON

   If one of the entries is incorrect (shown in red), carry out a fault finding exercise in accordance with the notes in section "Fault removal" p. 86.
5. Start the measurement.
   - Click on [START MEASUREMENT] or open the menu command MEASUREMENT ▸ START MEASUREMENT.
     The window MEASUREMENT START opens.
   - Enter the sample ID and, if applicable, a name for the analysis table. You can also enter the dilution, sample type, unit and remarks.

With [START ▶] open the window MEASUREMENT.
- Start the measurement by clicking the button [START F2] and follow the instructions of the control and analysis software.

  ✓ At the end of the measurement the results appear in the analysis report or in the selected analysis table.

6.5.2 Measurement with sampler

ATTENTION

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

1. In the window SYSTEM STATE select sample supply with sampler by clicking the button [SAMPLER].
   ✓ This is followed by the initialization of the analyzer.
2. In the window SYSTEM STATE check the following entries:
   - Optical bench - OK
   - Gas flow - OK
   - UV lamp - ON

   If one of the entries is incorrect (shown in red), carry out a fault finding exercise in accordance with the notes in section "Fault removal" p. 86.
3. Using the menu command METHOD ▸ NEW create a new method or load an existing method.
   To do so open via the menu command METHOD ▸ LOAD the database window METHOD SELECTION, highlight the desired method and confirm the selection by clicking the button [OK].
4. Fill the sample cups with the measuring liquid and place them onto the sample tray.
5. Only for NPOC measurements with AS vario or EPA sampler:
   Fill the acid cup with H_2SO_4 (c = 2 mol/l) and place the cup into the acid position of the sample tray:
   For the AS vario autosampler:
   - Position 14 on sample tray 20
   - Position 42 on sample tray 52
   - Position 55 on sample tray 72
   - Position 85 on sample tray 100
   - Position 131 on sample tray 146

   For the EPA sampler: Position 54 on sample tray 64
6. Start the measurement.
   - Click on [START MEASUREMENT] or open the menu command MEASUREMENT ▸ START MEASUREMENT. The window MEASUREMENT START opens.
   - In the window MEASUREMENT START enter a name for a new analysis table or select an existing analysis table with [EDIT].
   - With [START ▶] open the window CURRENT SAMPLE DATA.
   - Open an existing rack table or enter the sample name in the column SAMPLE ID in accordance with the assignment of the sample rack. You can also enter the dilution, sample type, unit and remarks.
   - With  release the samples.

- Confirm the entries with  .

✓ The rack table will be closed.

- A query follows whether the rack table should be saved. If you want to reuse the entries later, open the default window for saving files with [YES].
- Next the window MEASUREMENT opens. Start the measurement with [START F2] and follow the instructions of the control and analysis software.

✓ At the end of the measurement the results appear in the selected analysis table.

7 Maintenance and care

7.1 Maintenance intervals

Analyzer	
Maintenance task	**Maintenance interval**
Clean and maintain the device	weekly
Clean drip trays and reagent bottles	weekly and after filling
Inspect all hose connections for tight fit	weekly
Inspect the fastening screws for tight fit	monthly
Water traps	
Maintenance task	**Maintenance interval**
Check gas flow	daily
Exchange water trap	as required, but no later than after 6 months
Halogen trap	
Maintenance task	**Maintenance interval**
Check copper/brass wool for discoloration	daily
Replace depleted copper/brass wool	if half the copper wool is discolored black or the brass wool is discolored
TIC condensate container	
Maintenance task	**Maintenance interval**
Check for cracks and damages	3 months
Cleaning the TC condensation vessel	as required, but no later than after 12 months
Condensate pump	
Maintenance task	**Maintenance interval**
Check for leaks	3 months
Replace porous pump hose	as required, but no later than after 12 months
Syringe pump	
Maintenance task	**Maintenance interval**
Check for leaks	3 months
Clean the dosing syringe	as required, but no later than after 12 months
Reactor, UV lamp	
Maintenance task	**Maintenance interval**
Check the lamp intensity	12 months
Clean the UV reactor	as required

ATTENTION

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

ATTENTION

Ensure that all connections are gas-tight again after servicing:

- Do not insert the Fingertight screw connections twisted!
- Tighten all screw connections finger-tight!
- Check the system for leaks (see section "Checking the system for tightness" p. 84).

7.2 Adjustment and setup tasks

7.2.1 General notes for adjusting the autosampler

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

An adjustment of the sampler is necessary:

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

Adjusting the AS 10 and AS 21 autosamplers is described in the "Connecting add-on devices" section, p. 38.

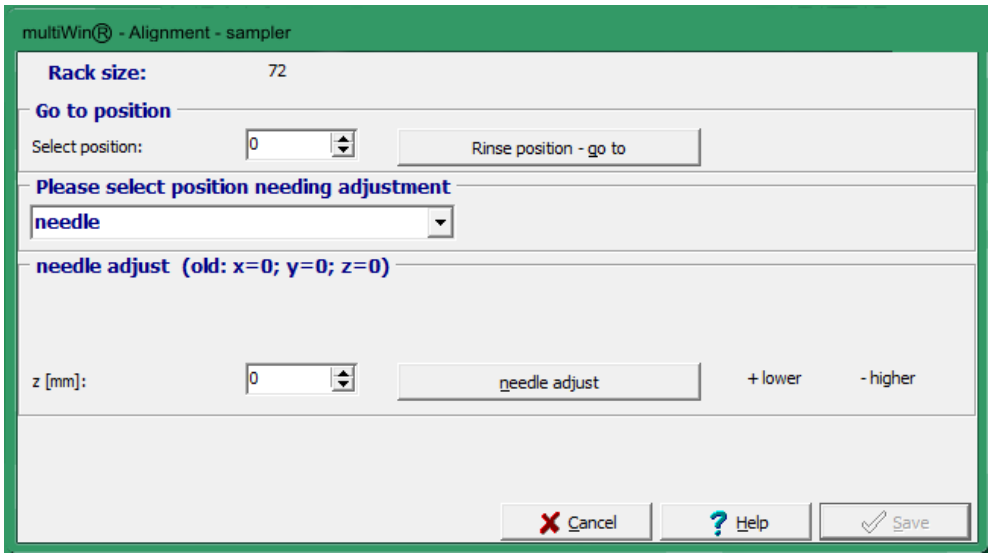
7.2.2 Adjusting the AS vario autosampler

ATTENTION

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

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

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

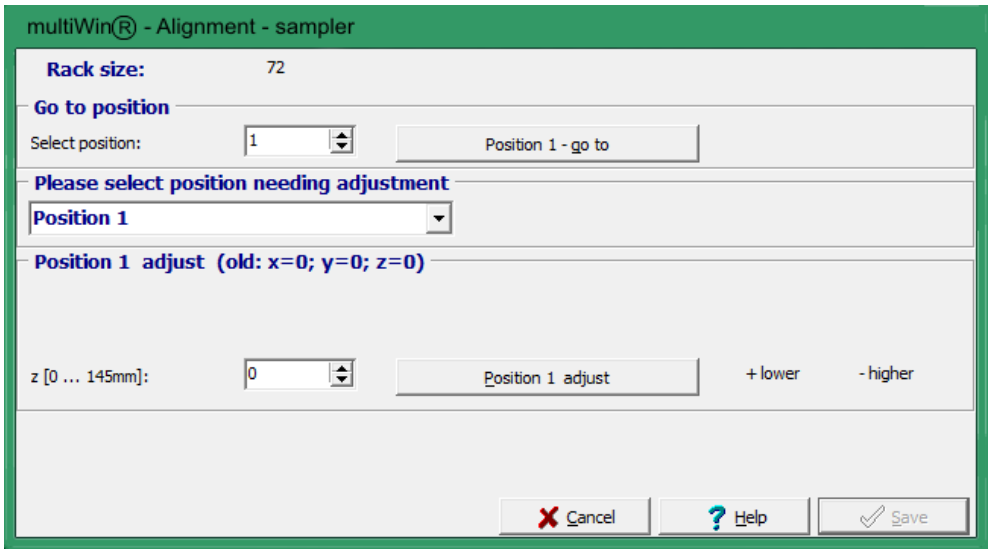


4. Increase or decrease the z values until the cannulas are positioned approx. 2 cm above the adjustment points and click the [NEEDLE ADJUST] button.
5. Align the cannulas with the two adjustment points by carefully bending them.

Fig. 36 Adjustment points on the sample tray

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

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



7. To adjust position 1, place a sample tube with magnetic stirrer onto the sample tray.
8. Increase or reduce the z values to align the rinse position or position 1.

   Adjust the height of the cannulas in the rinse position so that the cannulas immerse at least 1 cm into the rinse vessel.

   Adjust the height of the cannulas in position 1 to allow the stirrer free movement (distance about 5 mm).
9. Click the [RINSE POSITION ADJUST] or [POSITION 1 ADJUST] button.
   The autosampler will move to the new position. Repeat this step until the cannula position is optimal.
10. Click [SAVE].
    ✓ The adjustment values will be taken over.
11. Open the ALIGNMENT SAMPLER window again and click the corresponding button to move to the rinse position/position 1 again to check the alignment.

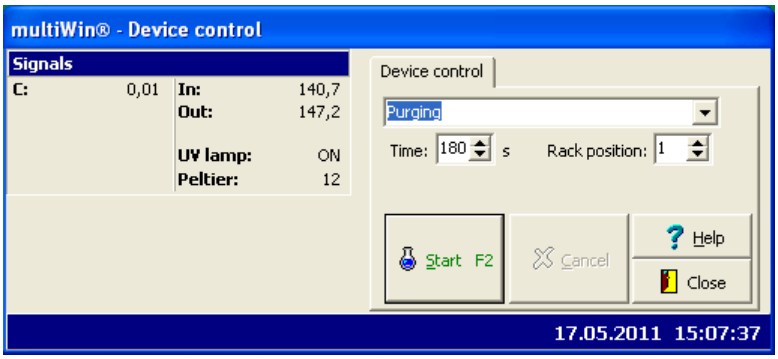
    Any position on the sample tray can be moved to for checking.

7.2.3 Adjust the NPOC purge flow

The NPOC purge flow has been preconfigured to approx. 100 ml/min. Dependent on the measuring task you can increase or reduce the NPOC purge flow via the NPOC needle valve. The needle valve NPOC is at the device front behind the left-hand door.

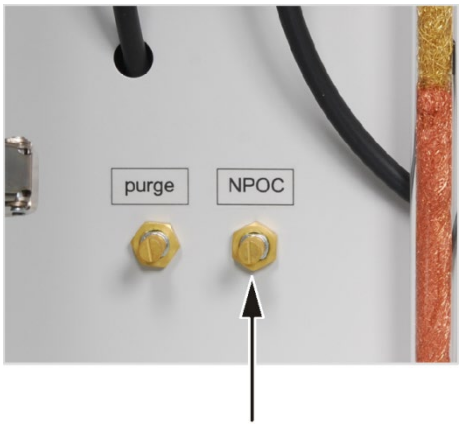
Adjust the NPOC purge flow as follows:

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



2. From the list field select the option PURGING.
3. For sample supplied with sampler:
   - Select the purging time in the field TIME between 1 and 900 seconds.
   - In the RACK POSITION field, select any position on the sample tray in which you want to monitor the purge flow.
   - Place a sample cup with ultrapure water onto this position.

   For manual sample supply:
   - Select the purging time in the field TIME between 1 and 900 seconds.
   - Insert the purging hose no. 10 into the cup filled with ultrapure water for which the purge flow is to be adjusted.



4. Click on the button [START F2].
5. Undo the adjustment screw at the NPOC needle valve.
6. Regulate the desired NPOC purge flow:
   - Increasing the NPOC purge flow - turn needle valve to the left.
   - Reducing the NPOC purge flow - turn needle valve to the right.
7. Relock the adjustment screw at the needle valve.

7.2.4 Adjusting the EPA sampler

ATTENTION

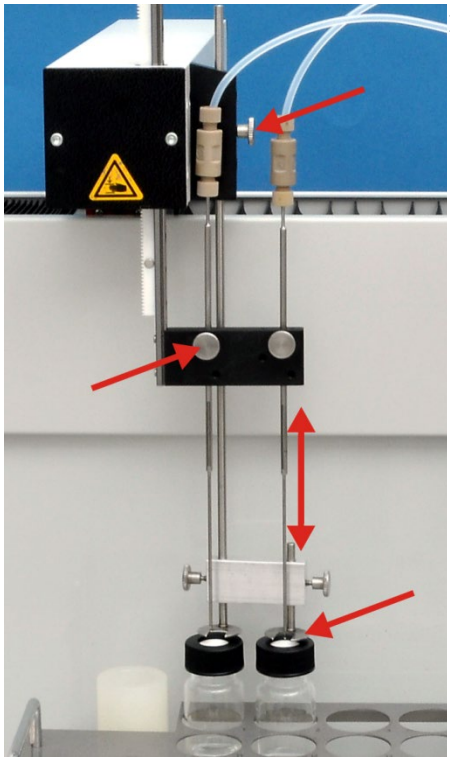
The cannulas can bend! Before adjusting the sampler undo the screw connections of the sample aspiration and purging cannulas!

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

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

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

For NPOC measurements, adjust the immersion depth for automatic acidification (z position). Adjust the cannula in the rinse position and test the adjustment values by automatically moving to the acid position. Make sure the cannula perforates the cover but does not enter the sample liquid.

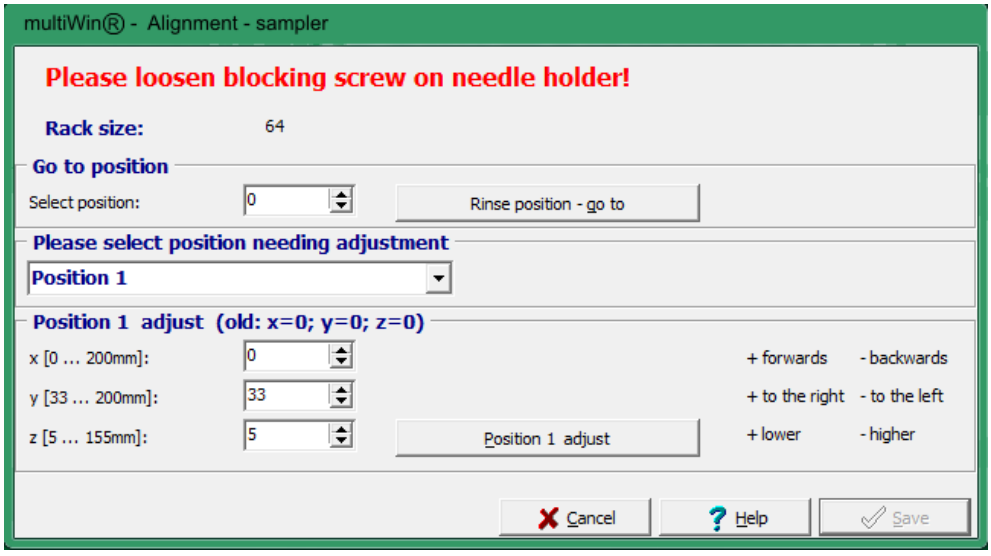


1. Install the holding-down clamps and sample aspiration cannulas in the cannula holder.

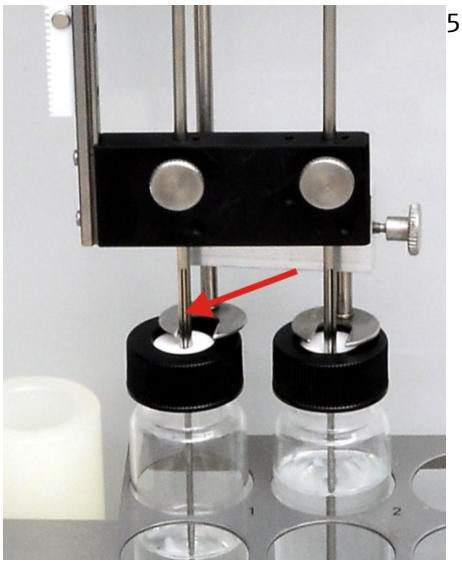
   Attention: The cannula can bend! Release the fastening screws of the cannula before performing the adjustment. Clamp the cannulas into the holder such that the cannula tip does not get immersed in the sample tube.

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

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



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



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

   The y value may not be smaller than 33 mm to ensure correct operation.

   - To adjust the z position, place the sample tube with screw closure and septum cap (e.g., EPA sample tube) into the sample tray.
     Adjust the special needle in the z direction until approx. 2 mm of the ventilation slot are visible above the septum.

     The ventilation slot must be above and below the septum, otherwise the pressure compensation within the sample tube cannot be guaranteed.
6. Adjust the rinse position.
   - Adjust the x and y positions until the cannula is at the center of the wash cup.
   - In the z direction the special cannula may only dip low enough for the ventilation slot to be visible at the top edge of the wash cup.
7. After each change of the x/y/z direction, click the [RINSE POSITION ADJUST] or [POSITION 1 ADJUST] button.
   The autosampler will then move to the new coordinates. Repeat this step until the cannula position is optimal.
8. Click [SAVE].
   ✓ The adjustment values will be taken over.
9. Open the ALIGNMENT SAMPLER window again and move to the selected position or any measuring position to check the alignment.

7.3 Replacing the water traps

ATTENTION

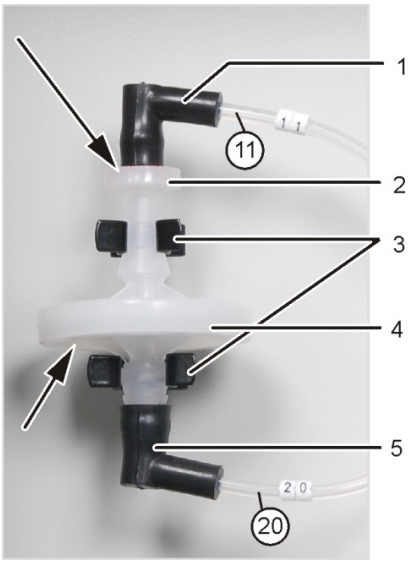
Replace the water traps dependent on the sample matrix, but no later than after 6 months.

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

Water traps on the front of multi N/C pharma UV

The two water traps (TC pre-filter, disposable retention filter) on the front of multi N/C pharma UV are installed between the cooling block and the halogen trap. They protect the MFM (mass flow meter) and the detector from aerosol and rising water.

These two water traps can be replaced in the switched-on state but not during a measurement.



1 FAST connector to hose no. 11
2 Disposable retention filter
3 Clamps
4 TC pre-filter (aerosol trap)
5 FAST connector to hose no. 20

1. Open the doors of the analyzer.
2. Pull the water traps out of the clamps (3).
3. Pull the FAST connectors (1) and (5) off the water traps.
4. Assemble the new water traps.

   The label "INLET" on the large water trap (aerosol trap) must point down and the label of the small water trap (disposable retention filter) must point up (arrows in Fig. on the right)
5. Connect the large water trap with the FAST connector to hose no. 20.
6. Connect the small water trap with the FAST connector to hose no. 11.
7. Press the water traps into the clamps on the equipment back-plate.
8. Check the system for leaks (see section "Checking the system for tightness" p. 84).
9. Close the front doors.

Water traps between the gasbox and the UV module

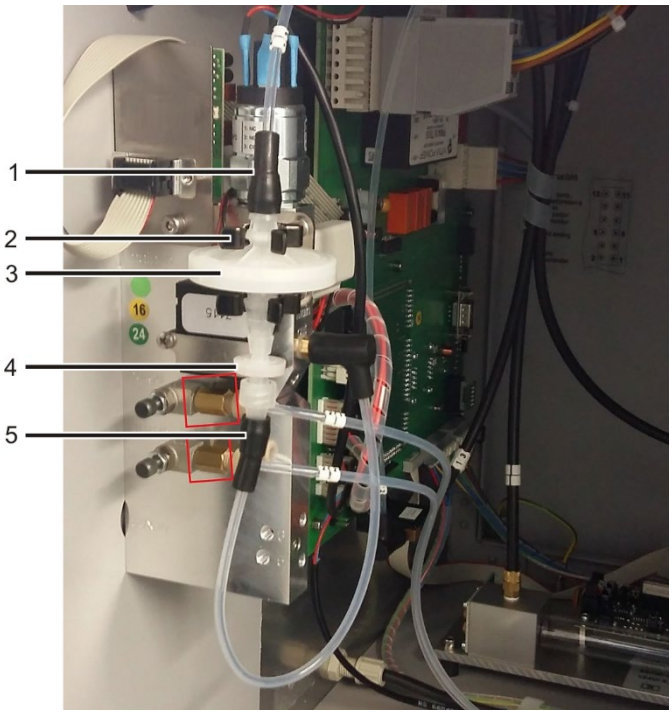
There are two water traps (prefilter and disposable retention filter) installed between the gas box and the UV module. Those two traps protect the gas box from aerosols or rising water in case of incorrect gas pressures. The left side panel of the analyzer must be opened to replace the water traps.

WARNING

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

CAUTION

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



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

Fig. 37 Water traps on the inside of the device, left side panel opened

1. Exit the control and analysis software multiWin with a backwash of the analyzer (see section "Switching off before longer periods of rest" on page 55).
2. Press the power switch to turn off the analyzer and remove the mains plug from the power outlet.
3. Remove the left side panel from the analyzer.
   Unscrew the four fastening screws; the screws are captive and remain in the panel.
   Disconnect the grounding conductor connection and put the side panel safely aside.
4. Pull the water traps off the two clamps on the gas box (2).
5. Pull the FAST connectors (1) and (5) off the water traps.
6. Assemble the new water traps.
7. The label "INLET" on the large water trap (aerosol trap) must face upwards and the label of the small water trap (disposable retention filter) must be directed downwards.
8. Use the FAST connector of the large water trap to connect it to hose no. 23 (UV reactor hose).
9. Use the FAST connector of the small water trap to connect it to the hose of the gas box.
10. Press the water traps into the clamps on the gas box.
11. Use the screws to refit the left side panel.
    Connect the grounding conductor connection to the left side panel.
    First screw in the screws at the bottom and then at the top. Tighten the screws in turn.
12. Insert the mains plug into the power outlet and press the main switch to turn on the analyzer.
13. Check the system for leaks (see section "Checking the system for tightness" p. 84).

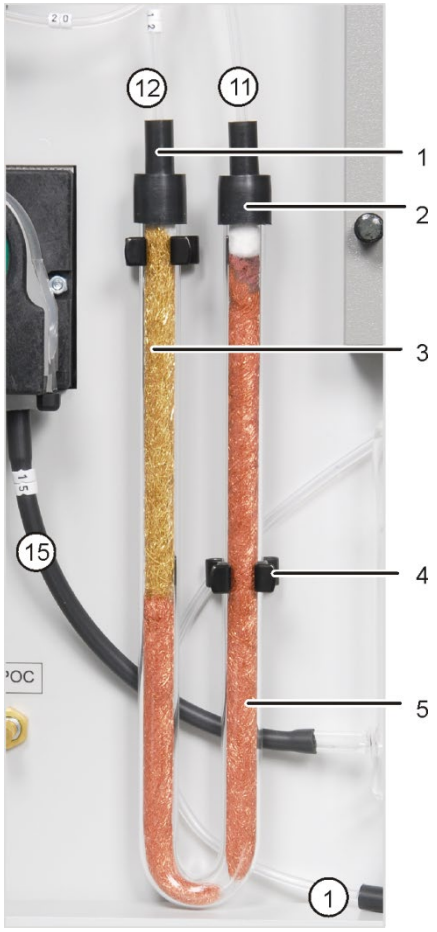
7.4 Replacing the halogen trap

ATTENTION

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

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

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



1 FAST connector no. 12 to the detector
2 FAST connector no. 11 to the water trap
3 Branch with brass wool
4 Clamp
5 Branch with copper wool

1. Open the doors of the analyzer.
2. Pull the U-shaped tube out of the clamps (4).
3. Pull the FAST connectors (1 & 2) off the halogen traps.
4. Pull out the depleted copper or brass wool from the U-tube with a tweezers or a small hook.
5. Check the U-tube for cracks.

   Only reuse a fully intact U tube!
6. If required, flush the U-tube with ultrapure water and leave to dry completely.
7. Fill the U-tube with new copper and brass wool using tweezers or a small hook.

   Replace the complete content of the U tube. When filling the halogen trap make sure that the copper and brass wool is not compacted too much and no larger empty spaces are created in the U-tube.
8. Cover the copper and brass wool with cotton wool.
9. Connect hose no. 11 (from the water trap) to the gas inlet branch with copper wool and hose no. 12 (to the detector) to the gas outlet branch with brass wool.
10. Press the halogen trap carefully into the clamps.
    Route the hoses no. 15 and no. 1 behind the halogen trap.
11. Check the system for leaks (see section "Checking the system for tightness" p. 84).
12. Close the doors of the analyzer.

7.5 Maintaining the UV reactor

- It is recommended to test the illumination of the UV lamp every 12 months to guarantee that the sample is fully digested.
- If the illumination of the lamp is insufficient, the UV reactor must be cleaned.
- If cleaning does not improve the performance of the lamp, the UV reactor must be replaced by the Analytik Jena GmbH customer service.

7.5.1 Checking the illumination of the lamp

The illumination of the lamp is tested by performing one TOC measurement with and another TOC measurement without sodium peroxodisulfate. The results of both measurements are used to calculate the quotient which is then multiplied by 100 % The UV lamp's illumination capacity is only sufficient if this ratio is between 85 % and 115 %.

The basis for this test is a sucrose standard solution (with c = 10 mg/l).

Method configuration

State	Liquid
Method	NPOC / with and without additional reagent
Determinations	min. 2, max. 3
Sample volume	5000 µl
NPOC purge time	300 s

Measurements

Measurement	Description	Results
1	Measurement without sodium peroxodisulfate, oxidation only by UV lamp	Surface integral SI_1
2	Measurement with sodium peroxodisulfate as an additional oxidant	Surface integral SI_2

Calculation

Quotient = SI_1 x 100 % / SI_2

If the quotient is above 115 %, a new standard and a new oxidant must be prepared and the test must be repeated.

If the quotient is below 85 %, the UV reactor may be contaminated which affects the efficiency of the UV radiation.

7.5.2 Cleaning the UV reactor

The UV reactor can be cleaned with the oxidizing reagent: $Na_2S_2O_8$ (with c = 80 g/l). It is not necessary to remove the UV reactor.

For cleaning, dip the sample aspiration cannula into the reagent bottle filled with $Na_2S_2O_8$ solution and start one measurement by hand (manual mode).

Method configuration

State	Liquid
Method	TC / with additional reagent
Determinations	min. 2, max. 3
Rinse cycles	1|0|0|0
Sample volume	20,000 µl
Rinse volume	2500 µl
Max. integration time	600 s

After completing the cleaning process, carry out additional purging measurements using ultrapure water in NPOC mode. Here too, use the maximum injection volume of 20,000 µl and 2-3 determinations to fully clean the reactor.

Recheck the illumination capacity of the lamp again after cleaning. If the cleaning does not improve the illumination, the UV reactor must be replaced by the customer service.

7.6 Cleaning the TC condensation vessel

CAUTION

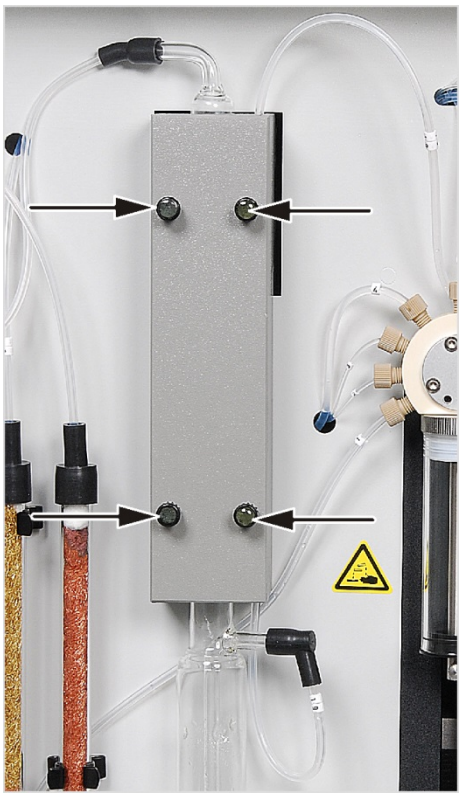
The TIC condensate container contains phosphoric acid! Phosphoric acid is irritating for eyes, skin and the mucous membrane!

Always wear goggles and protective gloves when handling concentrated phosphoric acid! Rinse the affected skin with water immediately.

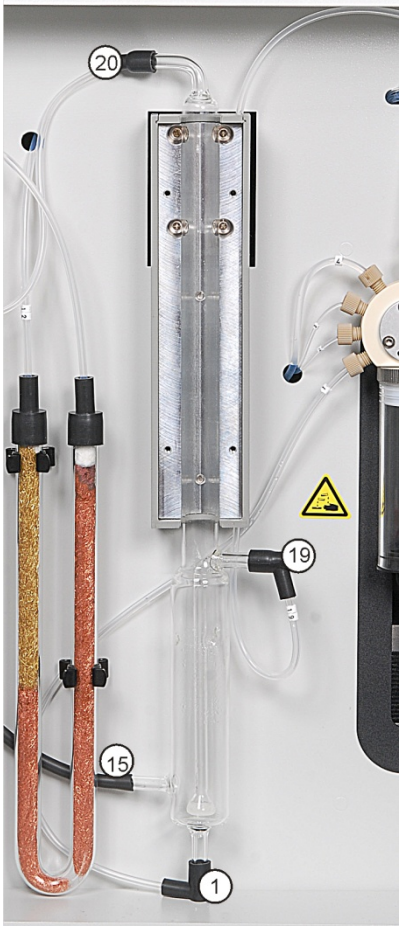
Visually inspect the TIC condensate container regularly for deposits. A cleaning is only required when the purging of the sample is not ensured anymore.

Remove the TIC condensation vessel as follows and clean it:

1. Exit the control and analysis software multiWin.
2. Open the doors of the analyzer.
3. Remove the hoses from the ultrapure water bottle, sample bottle and reagent bottle and wipe them with a clean paper towel.
4. Remove the reagent bottles and drip trays from the analyzer.



5. Release the 4 knurled head screws (arrows) at the cover of the cooling block.
6. Remove the cover and the metal plate below.
7. Remove the TIC container from the tray and pull the hoses out of the FAST connectors. Pull the FAST connectors off the connections of the TIC condensate container.
8. Check the TIC condensation vessel for deposits and cracks.
9. If required, rinse the TIC condensation vessel with ultrapure water.



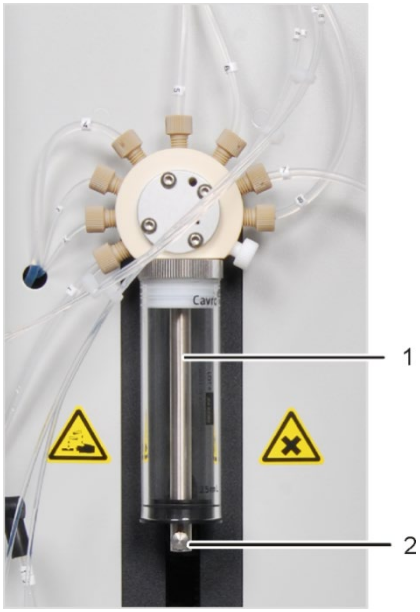
10. Attach the hoses as shown in the adjacent figure:
    - Push waste hose no. 15 at least 1 cm onto the bottom lateral connection of the TIC condensate container.
    - First push hoses no. 1, 19 and 20 into the FAST connectors, then plug the connectors onto the corresponding connections of the TIC condensate container.

    Route hoses number 1 and 15 behind the halogen trap as shown in the figure on the right.
11. Insert the TIC condensate container into the cooling block, place the metal plate and cover on it and attach the cover of the cooling block with the four knurled head screws.
12. Place the drip trays and reagent bottles into the analyzer.
13. Insert the hoses into the ultrapure water bottle, sample bottle and reagent bottles.

7.7 Cleaning and replacing the syringe pump

Replace or clean the syringe pump as follows:

1. Open the doors of the analyzer.
2. Drain the syringe pump via the software.
   - Using the menu command INSTRUMENT ▸ DEVICE CONTROL open the window of the same name.
   - Select the option CHANGE SYRINGE and click on [F2 START].
   - ✓ The syringe is drained and moved into the replacement position.
3. Remove the hoses from the ultrapure water bottle, sample bottle and reagent bottle and wipe them with a clean paper towel.
4. Remove the reagent bottles and drip trays from the analyzer.



5. Unscrew the knurled head screw at the drive rod (2).
6. Unscrew the glass cylinder (1) from the valve head.
7. Dismantle the glass cylinder and piston and clean them with ultrapure water.
8. Reassemble the glass cylinder and piston and screw the glass cylinder to the valve head.
9. Attach the piston with the screw (2) to the drive rod.
10. Place the drip trays and reagent bottles into the analyzer.
11. Insert the hoses into the ultrapure water bottle and reagent bottle
    - ultrapure water: hose no. 5
    - reagent bottle with H_3PO_4: hoses no. 2 and A
    - reagent bottle with $Na_2S_2O_8$: hoses no. 3 and B
    - sample intake cannula: hose no. 6
    - sample purge cannula: hose no. 10

7.8 Removing and replacing the pump hose

CAUTION

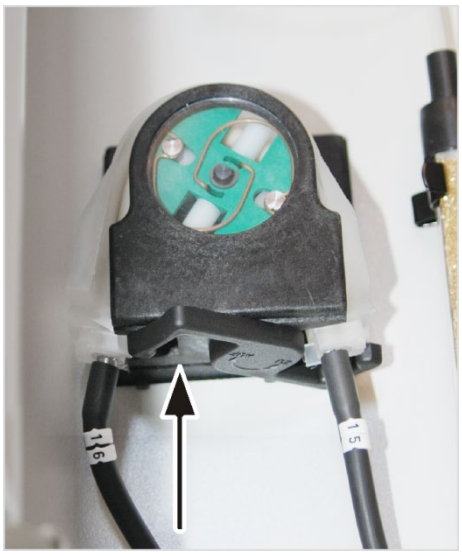
The pump hose contains phosphoric acid! Phosphoric acid is irritating for eyes, skin and the mucous membrane!

Always wear goggles and protective gloves when handling concentrated phosphoric acid! Rinse the affected skin with water immediately.

Inspect the pump hoses of the condensate pump every 3 months.

Condensate pump

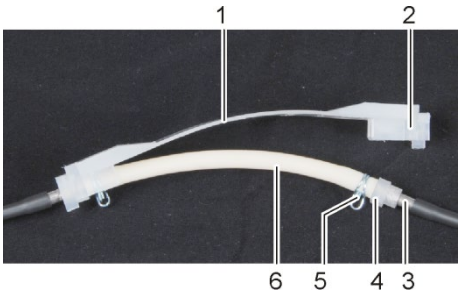
Remove the pump hose of the condensate pump as follows and inspect it for leaks:



1. Open the doors of the analyzer.
2. Push the bracket on the condensate pump (arrow) to the left.
3. Pull the hoses no. 16 and no. 15 off the connections.
4. Remove the conveyor belt with the pump hose from the pump body.
5. Check the pump hose and the connections on excessive wear and cracks.

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

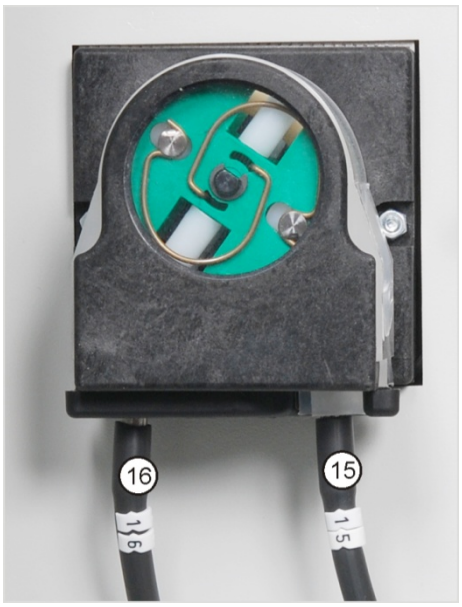
   If the pump body and roller carrier are heavily corroded, please contact the service department of Analytik Jena GmbH.



8. Push the faultless or new pump hose back into the conveyor belt.

   During installation the hose clamps must be rotated downwards. Push the hose guide into the groove on the conveyor belt.

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



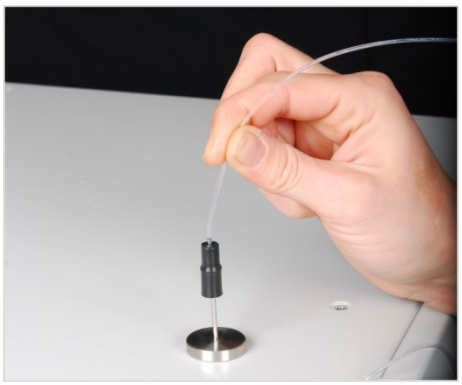
9. Push hose no. 15 and hose no. 16 back onto their adapters.
10. Position the conveyor belt around the pump body.
11. Press the conveyor belt down with one hand and turn the bracket to the right with the other hand until it locks into place.
12. Check the system for leaks (see section "Checking the system for tightness" p. 84).

7.9 Replacing the hose connections

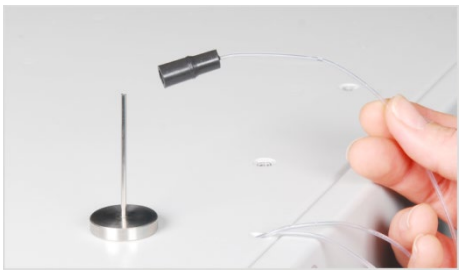
Check the hose connections regularly for leaks. Remove and replace faulty hoses and hose connections. Check the system for leaks (see section "Checking the system for tightness" p. 84).

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

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

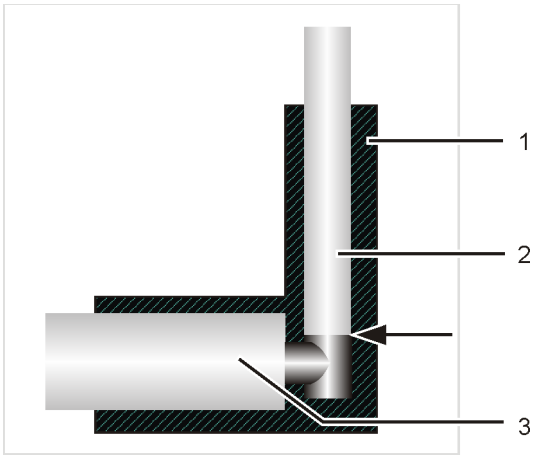


2. Thread the hose into the cannula of the threading aid.



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

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

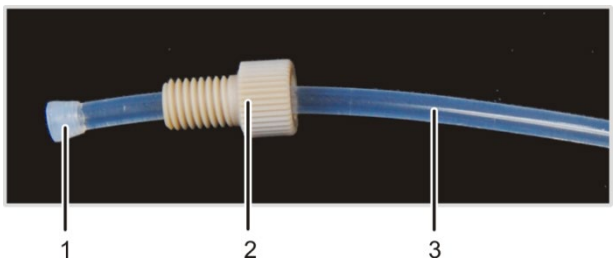


1 angled FAST connector
2 hose
3 connection adapter

Fig. 38 Angled FAST connector with connected hose

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

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



1 Conical nipple
2 Banjo bolt
3 hose

Fig. 39 Replacing the Fingertight connection

7.10 Checking the system for tightness

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

1. Switch on the analyzer multi N/C pharma UV.
2. Open the carrier gas supply at the pressure reducer.
3. Start the control and analysis software multiWin.
4. Check the flow indication in the window SYSTEM STATE:

- In (inlet flow): 140 ml/min
- Out (outlet flow): 140 ml/min (± 10 ml/min)

ATTENTION

If the outlet flow is significantly below the inlet flow of 140 ml/min, all connection points must be reinspected.

8 Fault removal

8.1 General information

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

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

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

Before starting a measurement a flow control is always carried out. A flow error is registered as soon as the actual flow differs ±10 ml/min from the target flow. In case of a flow error the UV lamp switches off.

In standby mode the measuring gas flow is lowered below the minimum flow, the UV lamps switches off for safety reasons.

For fault analysis it is possible to record log files. The recording of log files should be enabled after consultation with the Analytik Jena GmbH service department for specific faults. The log files are stored in the directory ...\multiWin\LOG. For fault diagnosis the complete directory ...\multiWin\LOG must be emailed to the Service department of Analytik Jena GmbH (see Service address on the inside front cover).

The following files can be generated and saved:

- multiWin_LOG.*:
  - Log file for error messages
  - always generated automatically
- multiWin_COM.*:
  - Log file for the recording of the interface commands
  - activation by program start with command START ▸ PROGRAMS ▸ MULTIWIN ▸ MULTIWIN WRITE MULTWIN_COM.TXT
- multiWin_ADU.*:
  - Log file to monitor the NDIR detector
  - generated automatically
  - With the menu command INSTRUMENT ▸ COMPONENT TEST open the window COMPONENT TEST ▸ tab OPTICAL BENCH and enable the field SAVE VALUES via (✓).

ATTENTION

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

8.2 Error messages in multiWin

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

7 8 9	**COM 2 not found** **COM 3 not found** **COM 4 not found**	
	Cause	**Remedy**
	■ problems with internal hardware	■ switch analyzer off/on
10	**Gas pressure error**	
	Cause	**Remedy**
	■ counterpressure in the analyzer system too great: carrier gas supply is automatically interrupted to protect the analyzer; flow indication MFC (IN) approx. 0 ml/min	■ search for and replace component causing the gas pressure error
	■ water trap clogged	■ renew the water traps and check whether the gas pressure error occurs again ■ see also p. 93
12	**Incorrect version number**	
	Cause	**Remedy**
	■ the versions of multiWin and the software of the internal computer do not match	■ update as appropriate
13	**No connection to the autosampler**	
	Cause	**Remedy**
	■ sample not switched on ■ connection cable not connected or faulty	■ switch on sampler and initialize analyzer ■ check connection cable
15	**Flow error in the window System state under gas flow** **Flow leak shown in red and** **in the window Measurement: values of IN and OUT shown in red** **Flow indication IN: 140 ml/min** **Flow indication OUT: < 130 ml/min**	
	Cause	**Remedy**
	system is leaking ■ UV reactor faulty (fractures at the connections) ■ TIC condensate faulty (fractures at the connections) ■ TIC condensate container connections leaking ■ connections at the water trap system loose (after installing water traps, installing halogen trap) ■ aerosol/water trap clogged ■ hose pump leaking	■ check glass components, replace if faulty ■ FAST connector at TIC condensate container, water traps ■ replace water traps (see error aerosol/water trap clogged) ■ check hose pump, replace pump hose if necessary ■ initialize analyzer
20 21 22 26	**no connection to optics (NDIR)** **CRC error optics** **status error optics** **optics error; incorrect command return**	
	Cause	**Remedy**
	■ communication error	■ initialize analyzer
	■ NDIR detector faulty	■ inform Service

24	**Optics error, analog values outside of range**	
	Cause	**Remedy**
	■ the analog values of the detector are outside the working range	■ check the quality of the carrier gas
	■ NDIR detector faulty	■ inform Service
40	**no connection to the syringe pump**	
	Cause	**Remedy**
	■ no communication between analyzer and syringe pump	■ initialize analyzer ■ switch off PC, switch back on and initialize analyzer
81	**UV cover open**	
	Cause	**Remedy**
	■ contact at UV cover not closed (e. g. after replacing the UV module)	■ close cover
84	**Communication error in temperature controller**	
	Cause	**Remedy**
	■ communication error	■ inform Service
111	**Rotator error**	
	Cause	**Remedy**
	■ Drive incorrectly positioned, e.g. jammed ■ Drive faulty	■ Initialize analyzer ■ If the error cannot be corrected, contact Service
112	**Swivel drive error**	
	Cause	**Remedy**
	■ Drive incorrectly positioned, e.g. jammed ■ Drive faulty	■ Initialize analyzer ■ If the error cannot be corrected, contact Service
113	**Lifting drive error**	
	Cause	**Remedy**
	■ Drive incorrectly positioned, e.g. jammed ■ Drive faulty	■ Initialize analyzer ■ If the error cannot be corrected, contact Service
114	**Rack detection error**	
	Cause	**Remedy**
	■ Sample tray not positioned correctly	■ Position the sample tray again and make sure it clicks into place ■ Initialize analyzer
115	**Wrong rack**	
	Cause	**Remedy**
	■ Wrong sample tray set in the software	■ Check settings in the software (see section 5); if necessary, set a different sample tray

116	**Unknown sampler command**	
	Cause	**Remedy**
	■ Communication error	■ Contact Service
201	**Restart the internal program**	
	Cause	**Remedy**
	■ overvoltage ■ short-term power failure	■ initialize analyzer
401 402 403 404	**Syringe pump initialization** **Syringe pump: invalid command** **Syringe pump: invalid operand** **Syringe pump faulty command sequence**	
	Cause	**Remedy**
	■ communication error	■ initialize analyzer
	■ syringe pump faulty	■ inform Service
407	**Syringe pump: syringe pump not initialized**	
	Cause	**Remedy**
	■ analyzer not yet initialized after switching on ■ reset the syringe pump	■ initialize analyzer
409	**Syringe pump: pump sluggish**	
	Cause	**Remedy**
	■ clogging of a hose line (6) or (7)	■ search for cause and remedy fault ■ replace hose line - if necessary remove hose line and rinse with ultrapure water, then reinstall ■ initialize analyzer
	■ syringe pump faulty	■ inform Service
410	**Syringe pump: valve sluggish**	
	Cause	**Remedy**
	■ syringe pump faulty ■ valve broken	■ inform Service
411 415	**Syringe pump: pump step not permitted** **Syringe pump: invalid command**	
	Cause	**Remedy**
	■ communication error	■ initialize analyzer
	■ syringe pump faulty	■ inform Service
MESSx	**Analyzer error: MESSx measurement is cancelled**	
	Cause	**Remedy**
	■ equipment fault	■ initialize analyzer ■ check window *System state* after error removal ■ search for equipment fault and remove error

8.3 Status errors – indications in the window SYSTEM STATE

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

Error indication	
Flow indication IN: 140 ml/min **Flow indication OUT: < 130 ml/min or > 150 ml/min**	
Cause	**Remedy**
■ MFM (mass flow sensor) faulty	■ check flow with external mass flow sensor to confirm error if possible ■ inform Service
■ halogen trap filling used up	■ check halogen trap
Flow indication IN: < 140 ml/min or fluctuating **flow indication OUT : < 130 ml/min**	
Cause	**Remedy**
■ no carrier gas ■ hose line leaking	■ turn on carrier gas at the pressure reducer ■ search for and remedy leak
■ preliminary pressure at the carrier gas supply too low	■ set carrier gas preliminary pressure to 4 to 6 bar
■ pressure switch in the analyzer has tripped - simultaneous error message in multiWin "gas pressure error	■ see gas pressure error (error code 10) on page 88
■ MFC faulty	■ inform Service
Flow indication IN: 140 ml/min **Flow indication OUT: > 150 ml/min**	
Cause	**Remedy**
■ Peltier cooling insufficient (simultaneous error message temperature out of range below Peltier temperature)	■ check from the top at the TIC condensate container whether cooling takes place (formation of condensate at the cooling block indicates that the cooling is working)
■ MFC faulty	■ inform Service
Flow indication IN: 0 ml/min **Flow indication OUT: 0 ml/min**	
Cause	**Remedy**
■ a hose line is clogged up	■ replace clogged hose line ■ if necessary remove and rinse clogged hose line, then reinstall
■ gas flow interrupted	■ check connection
■ no method loaded	■ load method

Values of the NDIR detector below opt. bank indicated in yellow	
Cause	**Remedy**
■ The analog values of the detector are outside the working range. The ADU values can be viewed in the control and analysis software multiWin with the menu command **Instrument / Component test** on the tab ***Optical Bank***.	■ check halogen trap and replace if necessary ■ consult regarding the application with Analytik Jena GmbH about specific application rules for difficult sample matrix Measurements are still possible, but it should be pointed out to the user that the ADU values of the detector are leaving the optimum range
Temperature out of range, the Peltier value is marked in red in the window Measurement under Signals	
Cause	**Remedy**
Peltier cooling insufficient	■ inform Service

ATTENTION

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

8.4 Equipment faults and analytical problems

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

Error	
Initialization does not complete	
Cause	**Remedy**
■ no flow can be detected at the system outlet	■ open pressure reducer at the carrier gas bottle N_2/Ar ■ check that system (gas path) has been installed
UV lamp does not ignite during analyzer initialization	
Cause	**Remedy**
■ measuring gas flow outside the range of 130 to 150 ml/min	■ check flow, see "Status errors – indications in the window *System state*" p. 91
UV lamp switches off during operation	
Cause	**Remedy**
■ measuring gas flow drops during the pauses between measurements below the minimum flow, UV lamp switches off for safety reasons	■ check flow, see "Status errors – indications in the window *System state*" p. 91
■ lamp does not ignite even if the flow is ok, UV lamp faulty	■ notify service

Water traps clogged	
Cause	**Remedy**
■ Lifetime exceeded (renewal after 6 month recommended, matrix dependent) ■ Measuring of samples with strong aerosol generation ■ Peltier cooling insufficient – simultaneous error message in the window ***System state*** – temperature out of range	■ Replacement of the water traps (see section "Replacing the water traps" p. 74) ■ inform Service
Minimum sample volume > cup volume	
Cause	**Remedy**
■ sample volume selected too large ■ number of measurements too high	check configuration in the method: ■ sample volumes ■ wash volume ■ adjust number of detections (repeat measurements) to the cup volume
Rinse water insufficient (for sample supplied with sampler)	
Cause	**Remedy**
■ rinse reservoir insufficient	check configuration in the method: ■ reduce rinse volume ■ reduce number of backwash cycles
Scattering measured values	
Cause	**Remedy**
■ metering faulty	■ check metering ■ bubble free sample drawing ■ check that sufficient sample is available
■ metering syringe leaking	■ install new metering syringe
■ added reagents unstable	■ bubble free drawing of the reagent(s) ■ consider blank value ■ modify purge flow for reagents ■ check that sufficient reagents are available ■ insert hose deep enough into the container
■ sensitive samples	■ prevent introduction of CO_2 or organic vapors from the ambient air ■ cover sample cups on sampler with aluminum foil ■ for manual measurement do not aerate the head room of the sample container with N_2/Ar ■ check the environmental conditions ■ remove the source of interference
drift NDIR basic	
■ unsuitable integration criteria ■ measurement is canceled too early	■ check the settings ■ increase the maximum integration time
■ inhomogeneous sample matrix	■ filter samples prior to analysis ■ notify application of Analytik Jena GmbH (stir sample, use sampler with stirrer)

Sample is not drawn up without air bubbles	
Cause	**Remedy**
■ leaks in the sample intake path	■ check connections between cannula and hose and tighten any loose connections
■ sample intake cannula clogged	■ remove the cannula and clean in an ultrasonic bath ■ replace cannula
■ metering syringe leaking ■ PTFE sealing lips of the plunger are damaged	■ remove and check metering syringe ■ replace metering syringe
incomplete metering in reactors	
Cause	**Remedy**
■ leaks in the metering path	■ check connections and tighten any loose connections
Carry-over	
Cause	**Remedy**
■ insufficient syringe rinse	■ rinse the metering syringe with sample before the next injection: under METHOD ▸ EDIT in the tab METHOD enter 3 for rinse cycles for the first measurement, for all other measurements rinsing is generally not required, enter 0
Unusual peak shape	
Cause	**Remedy**
■ incomplete digestion	■ add reagent ■ reduce sample volume ■ dilute samples
Leaking condensate pump	
Cause	**Remedy**
■ leaking hose connections ■ defect pump hose	■ replace the pump hose
Control lamps at the analyzer do not illuminate	
Cause	**Remedy**
■ error in the power supply or in the electronics	■ check the electrical connections ■ check the power supply of the lab
■ equipment fuse faulty	■ inform Service

9 Transport and storage

9.1 Transport

9.1.1 Preparing the analyzer for transport

CAUTION

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

ATTENTION

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

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

Prepare the analyzer for transport as follows:

1. Rinse the phosphoric acid pump and corresponding hoses with ultrapure water and then drain these components.
2. Switch off the analyzer from the main switch and allow the equipment to cool down.
3. Disconnect the gas supply and unplug the mains plug from the mains outlet.
4. Undo all connections on the back of the analyzer.
5. Open the doors of the analyzer and remove the reagent bottles and drip trays and any other loose accessory components.
6. Pull the hoses off the connections at the halogen trap and press the halogen trap out of the clamps.
7. Remove and drain the TIC condensate container (see section "Cleaning the TC condensation vessel" p. 79).
8. Pack open hose ends in protective bags and secure them e.g. using adhesive tape.
9. Remove the cannulas from the hoses and insert the cannulas into the cannula packaging.
10. Carefully package the accessories, in particular protect glass components against breakage.

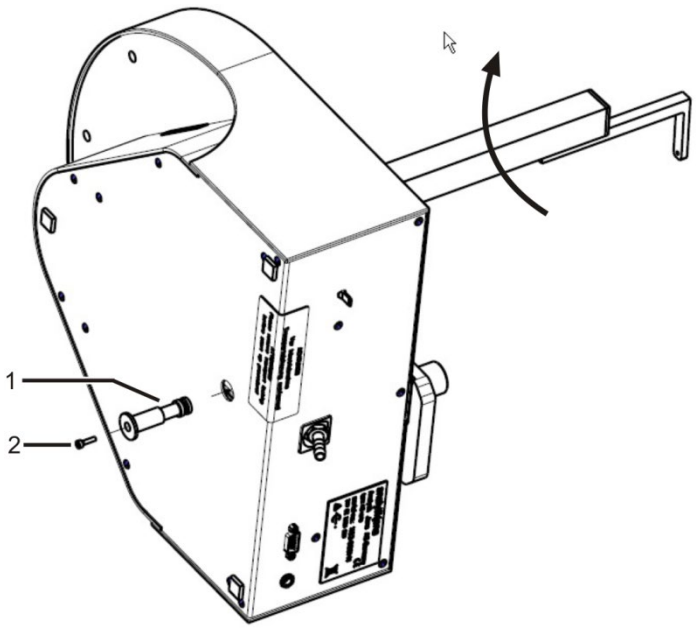
9.1.2 Transport notes

Observe the safety instructions in section "Safety instructions, transport and installation" p. 11. Transport the analyzer very carefully to prevent damage from impact or vibration. The analyzer should be transported in such a way that major temperature fluctuations are avoided and the formation of condensate is thus prevented.

9.1.3 Preparing the autosampler AS vario for transport

ATTENTION

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



1 Transport lock
2 Screw M3x12

Fig. 40 Transport lock on autosampler AS vario

1. Place the autosampler on the side as shown in Fig. 40.
2. Turn the autosampler arm clockwise to the stop.
   Then the drives are in the right position.
3. Push the red transport lock into the opening on the bottom of the autosampler as far as possible.
4. Bolt the screw (2 in Fig. 40) with the Allen wrench.

9.1.4 Moving the analyzer in the laboratory

CAUTION

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

Observe the following when moving the analyzer within the laboratory:

- Insufficiently secured components pose a risk of injury! Before moving the analyzer remove all loose components, in particular the reagent bottle with phosphoric acid and sodium peroxodisulfate.
- Disconnect all supply connections and any add-on devices from the analyzer.
- To prevent health damage the following must be observed when moving the analyzer in the laboratory (lifting and carrying):
  - For reasons of safety 2 persons are required to transport the analyzer and must position themselves on both sides of the equipment.
  - Because the analyzer does not feature any handles, firmly grip the device from the bottom and make sure prior to simultaneous lifting the device that the sensitive components at the front are protected by the closed doors.
- Observe the guide values and adhere to the legally mandated limits for lifting and carrying without auxiliary means!
- For the setup at the new location observe the notes in section "Site requirements" p. 36.

9.2 Storage

ATTENTION

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

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

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

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

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

9.3 Recommissioning after transport or storage

9.3.1 Assembling the analyzer after transport or storage

When positioning the analyzer observe the notes in section "Site requirements" p. 36.

1. Carefully remove the basic device, accessories and any add-on devices from the transport packaging. Do not damage the transport packaging!
2. Position the analyzer at the intended location.
3. Remove the adhesive tape from the doors and side walls. Open the doors.
4. Remove all remaining adhesive tape and protective bags.
5. Install the halogen trap and the water traps (the water traps are connected to the halogen trap) (see sections "Replacing the halogen trap" p. 77 and "Replacing the water traps" p. 74)
6. Install the TIC condensate container (see section "Cleaning the TC condensation vessel" p. 79).
7. Close the doors of the analyzer.

9.3.2 Connecting the analyzer

CAUTION

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

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

Only use the IEC connection cable included in the scope of delivery for the connection to the mains supply (VDE label, 1.5 m long). Extensions of the supply cable are not permitted!

ATTENTION

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

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

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

Fig. 41 Mains connection and gas connections at the multi N/C pharma UV

1 Holder for mains fuse "FUSE"
2 Main switch to switch the analyzer on and off "power switch"
3 Mains connection "main plug"
4 Gas connection "analyte"
5 Gas connection "pump"
6 Connection "internal"
The connections "analyte" and "internal" are linked via a hose bridge
7 Connection for inert gas "N_2 nitrogen"
8 Connection of the neutral conductor at the sampler
9 Waste connection "waste"
10 RS 232 interface for HT module "HT"
11 RS 232 interface for the sampler "sampler"
12 USB port for PC

Make the mains connection as follows:

1. Connect the low-heat connection cable to the mains connection at the rear of the analyzer (3 in Fig. 41 p. 99).
2. Connect the mains plug of the low-heat connection cable to a grounded socket.

Connecting the gas supply

ATTENTION

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

Make the carrier gas connection as follows:

3. Connect the connection hose supplied to the pressure reducer of the gas supply and the gas connection " N_2 nitrogen " on the equipment backplate (7 in Fig. 41 p. 99).
4. Set the preliminary pressure at the pressure reducer to between 4 and 6 bar.

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

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

Connecting accessories

Connect the reagent bottle and accessory components as follows:

5. Connect the waste hose to the "waste" connection at the analyzer backplate and lead the loose end into a suitable waste container or drain.
6. Open the front doors and place the reagent bottles with drip trays into the analyzer.
7. Connect the following hoses:
   - ultrapure water: hose no. 5
   - reagent bottle with H_3PO_4: hoses no. 2 and A
   - reagent bottle with $Na_2S_2O_8$: hoses no. 3 and B
   - sample intake cannula: hose no. 6
   - sample purge cannula: hose no. 10
8. Position any add-on devices at the intended location and connect them. Observe in this regard the user manuals of the add-on devices.

10 Disposal

10.1 Waste water

Waste water arises during the ongoing analysis operation of the multi N/C pharma UV. This water contains sulfuric acid, diluted phosphoric acid and sample, depending on the measurement mode.

The neutralized (if necessary) waste must be brought to the appropriate waste disposal center for correct disposal according to the appropriate legal guidelines.

10.2 Halogen trap

The halogen trap contains copper. Contact the responsible institution (authority or waste disposal company). There you will receive the information regarding recycling or disposal.

10.3 Analyzer

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

10.4 UV module

The UV module is equipped with a low-pressure mercury vapor lamp. For disposal, remove the UV module from the analyzer (see section "Removing the UV module"). Dispose of the UV module in accordance with the national regulations for lamps that contain mercury.

10.4.1 Removing the UV module

WARNING

Lethal electrical voltages may occur in the UV module of the analyzer! Touching the UV module with the device switched on can be fatal!

Before opening the right side panel, press the main switch to turn off the analyzer and remove the mains plug from the power outlet!

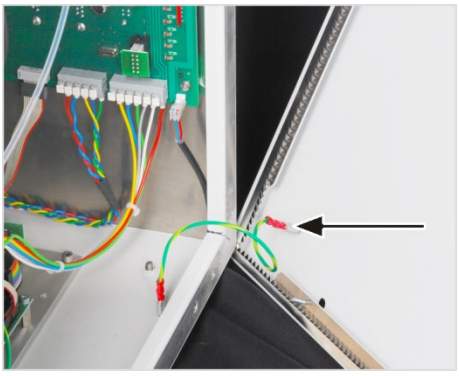
CAUTION

There is a risk of burning! The UV reactor remains hot immediately after switching off the analyzer!

Wait at least 30 minutes for the UV reactor to cool down before removing the UV module!

Preparation

1. Exit the control and analysis software multiWin. Press the main switch to turn off the analyzer and remove the mains plug from the power outlet.
2. Turn off the autosampler, unplug the mains cable and the serial data cable from the autosampler and remove the autosampler.
3. Pull the hoses off the ultrapure water bottle, the reagent bottles and the sample vial, where applicable. Wipe the hoses.
4. Remove the reagent bottles and drip trays from the analyzer.



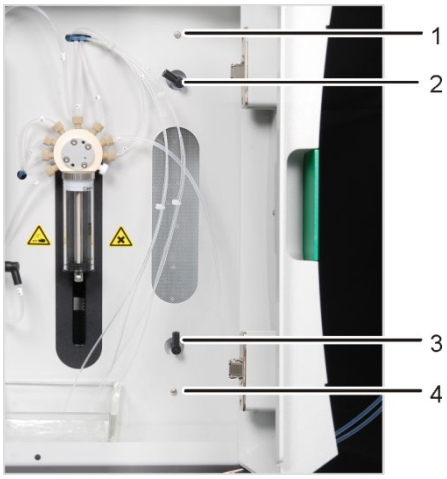
Removing the UV module

5. Remove the right side panel from the analyzer.

   Unscrew the four attachment screws. The screws are captive and remain attached to the panel.

   Disconnect the grounding conductor connection and put the side panel safely aside.

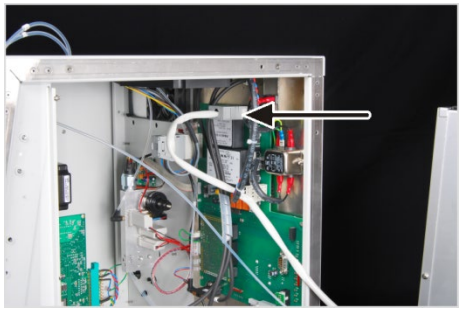
   ✓ The UV module is now accessible for removal.



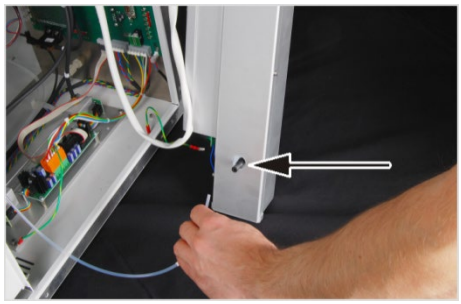
1, 4 Attachment screws
2 Connection for hose no. 19
3 Connection for hose no. 7

6. Detach the hoses no. 7 and 19 from the PTFE screw joints (2 and 3).

7. Hold onto the UV module inside the analyzer with your right hand and unscrew the attachment screws above and below the UV protective glass with your left hand (1 and 4).



8. Remove the complete UV module towards the back and the right.
9. Disconnect the plug-in connector from the connection in the analyzer.



10. Detach hose no. 23 from the PTFE screw joint on the UV module.
    ✓ The UV module is now removed.

11 Specifications

11.1 Technical data

General characteristics	
Designation/type	Analyzer multi N/C pharma UV
Dimensions	Basic device (W x H x D) (513 x 464 x 550) mm
Mass	approx. 28 kg
Procedural data	
Digestion principle	wet chemical through UV radiation or UVC radiation and $Na_2S_2O_8$
Measuring method	TC, TIC, TOC (differential method), NPOC
Industry standards	Pharm.Eur. <2.2.44>, USP <643>, JP <2.59>, FDA 21 CFR Part 11 Compliance
Temperature of the measuring medium	approx. 80 °C

Carbon detection	NDIR (coupled with VITA method)
Sample volumes	0.05 – 20 ml
Sample Feed	Flow injection
Gas supply	Nitrogen (minimum 5.0) Argon (minimum 4.6) Preliminary pressure 4 – 6 bar
Gas consumption: in total Measuring gas flow Purge flow	 approx. 15 l/h, dependent on the measuring mode 140 ± 10 ml/min approx. 100 ml/min
Control/analysis (control and analysis software multiWin)	Real-time graphics, status indication during analysis, graphical display of the measured results, result print-out
Electrical variables	
Connection	100-240 V AC, 50/60 Hz
Protection	T 4,0 A H (Use only original fuses of Analytik Jena GmbH)
Typical average power consumption	150 VA
PC interface	USB 2.0

Environmental conditions	
Temperature during storage	5 – 55 °C
Temperature during operation	10 – 35 °C
Humidity during operation	max. 90% at +30 °C
Humidity during storage	(10 – 30) % (use desiccant)
Air pressure	0.7 1.06 bar

Minimum equipment for the control and analysis unit

Operating system:	Windows 7 Professional or better
Processor:	Intel Core I3 or better
Working memory:	4 GB
Free hard disk space:	40 GB
Drive:	CD-ROM drive
Monitor resolution:	1028 x 768
Interfaces	USB 2.0

11.2 Standards and directives

Safety class and safety type

The analyzer belongs to protection class I.

The casing has protection class IP 20.

Device safety

The analyzer conforms to the safety standards

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

EMC compatibility

The analyzer has been checked for interference emission and resistance.

It meets the requirements for interference emission of

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

It meets the requirements of interference resistance of

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

Environmental compatibility

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

- ISO 9022-3
- ISO 9022-2

EU directives

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

Chinese directives

The analyzer contains regulated substances (according to directive SJ / T 11363-2011). Analytik Jena GmbH guarantees that these substances will not leak out during the next 25 years if the analyzer is used in accordance with its intended use and thus do not pose a threat to the environment or health within this period.