| Manufacturer | Analytik Jena AG  
| Konrad-Zuse-Str. 1  
| 07745 Jena · Germany  
| Phone + 49 3641 / 77 70  
| Fax + 49 3641 / 77 92 79  
| Email info@analytik-jena.de |
| Service | Analytik Jena AG  
| Konrad-Zuse-Str. 1  
| 07745 Jena · Germany  
| Phone + 49 3641 / 77-7407 (Hotline)  
| Email service@analytik-jena.de |
| General information | http://www.analytik-jena.com |
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1 Basic Information

1.1 User manual notes

The analyzer multi N/C pharma HT 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 action which occur in chronological order are numbered and combined into action units and furnished with the corresponding results.

Lists which are not in chronological order are shown as itemized lists, sub-listings as bullet points.

Safety notes are indicated by pictographs and signal words. The type and source of the danger are stated together with notes on preventing the danger. The meaning of the pictographs and signal words used is explained in section “Safety instructions” p. 6.

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

- Menu commands, buttons, options etc. are formatted in bold.
- Menu commands of a command sequence are separated by slashes ( / ), e.g. Method / Edit.
- Window names are shown in bold italics, e.g. Measurement window.
- Buttons are marked with square brackets, e.g. [Save].

1.2 Analyzer area of application

The analyzer multi N/C pharma HT is a device to determine the parameters TC, TOC, NPOC and TIC in aqueous samples. Detection is by way of thermocatalytic digestion in the presence of a special catalyst in accordance with the national and international standards. The analyzer has been designed in particular for pure and ultrapure water analysis and meets the requirements for water monography by the European Pharmacopeia Commission and the water monograph of the United States Pharmacopeia Convention (USP).

Complementing the analyzer with a Chemiluminescence detector permits the detection of TNb (total nitrogen bound).

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

In line with the device design in respect of measuring range, sample volumes and sample feeding the pure and ultrapure water analysis is the main area of application of the multi N/C pharma HT.
Pharmacy, medicine and biotechnology

Water for injection purposes (WFI), aqua purificata water for pharmaceutical purposes must not exceed a TOC limit of 500 µg/l, therefore the TOC content of this water must be controlled accordingly. The TOC devices used for this purpose must have a lower detection limit of at least 50 µg/l. With its specification the multi N/C pharma HT exceeds these requirements and is thus ideally suited for use in pharmaceutical laboratories, medical facilities etc.

Microelectronics and semiconductor technology

The TOC is an important parameter in the manufacture of semiconductors, because the water used in their production may only have very low TOC concentrations to guarantee a high product quality. Due to its sensitive measuring range the multi N/C pharma HT is also suited for this area of application.

Cleaning validation

The TOC parameter and, more recently, also the TNₜ parameter have become part of cleaning validation. The demands on the measuring range of the analyzer used are also very high in this case. The multi N/C pharma HT can be used here both for "final rinse" method and for the swab test.

1.3 Intended use

The analyzer multi N/C pharma HT must only be used for the methods for the detection of the total carbon and/or the total nitrogen content in aqueous samples as described in this user manual and for the detection of the total carbon content in small amounts of solid samples. Any other use is not as intended! Only the operator is liable for any damages that result from this.

In particular it is prohibited to use the analyzer to analyze flammable liquids or substances that could form explosive mixtures. No concentrated acids may be analyzed with the analyzer.

The device must only be used with oxygen as a carrier gas.

The operational safety of the analyzer multi N/C pharma HT 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 AG which are available from the customer service address stated above.

1.4 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 AG.

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 HT other than intended
- improper commissioning, operation and service of the analyzer
- modifications of the equipment without prior consultation with Analytik Jena AG
- 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
## Technical data

### General characteristics

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Specification</th>
</tr>
</thead>
<tbody>
<tr>
<td>Designation/type</td>
<td>analyzer multi N/C pharma HT</td>
</tr>
<tr>
<td>Basic device dimensions (W x H x D)</td>
<td>513 x 464 x 550 mm</td>
</tr>
<tr>
<td>Mass</td>
<td>approx. 28 kg</td>
</tr>
</tbody>
</table>

### Procedural data

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Specification</th>
</tr>
</thead>
<tbody>
<tr>
<td>Digestion principle</td>
<td>Thermocatalytic oxidation</td>
</tr>
<tr>
<td>Digestion temperature</td>
<td>up to 950 °C, depending on the catalyst</td>
</tr>
<tr>
<td>Catalyst</td>
<td></td>
</tr>
<tr>
<td>– for liquid samples</td>
<td>Pt(Al2O3) for pharma HT</td>
</tr>
<tr>
<td>– for swab test module</td>
<td>CeO2, special catalyst for multi N/C</td>
</tr>
<tr>
<td>Measuring method</td>
<td>TC, TIC, TOC (differential method), NPOC, TNb (optional)</td>
</tr>
<tr>
<td>Industry standards</td>
<td>Pharm.Eur. &lt;2.2.44&gt;, USP &lt;643&gt;, JP &lt;2.59&gt;, FDA 21 CFR Part 11 Compliance, Pharm.Eur. &lt;2.5.33, 7B&gt;, USP &lt;1047, 7.2&gt;</td>
</tr>
<tr>
<td>Carbon detection</td>
<td>NDIR (coupled with VITA method)</td>
</tr>
<tr>
<td>Nitrogen detection (optional)</td>
<td>CLD</td>
</tr>
<tr>
<td>Sample volumes</td>
<td>50 – 3000 µl</td>
</tr>
<tr>
<td>Sample Feed</td>
<td>Flow injection</td>
</tr>
<tr>
<td>Gas supply</td>
<td>Synthetic air (free of HC, free of CO2)</td>
</tr>
<tr>
<td></td>
<td>Synthetic/Purified air can be supplied from gas cylinders or after clean-up of pressurized air by a TOC gas generator.</td>
</tr>
<tr>
<td></td>
<td>Purity specifications to be met: CO2 &lt; 1 ppm</td>
</tr>
<tr>
<td></td>
<td>Hydrocarbons &lt; 0,5 ppm (as CH4)</td>
</tr>
<tr>
<td></td>
<td>Supply pressure: min. 5 bar (72 psi)</td>
</tr>
<tr>
<td></td>
<td>Provided flow rate: min. 300 ml/min</td>
</tr>
<tr>
<td></td>
<td>Oxygen (minimum 4.5)</td>
</tr>
<tr>
<td></td>
<td>Preliminary pressure 4 – 6 bar</td>
</tr>
</tbody>
</table>

### Gas consumption:

- **in total:**
  - Analyte gas flow: approx. 15 l/h, dependent on the measuring mode
  - Purge flow: approx. 120 ± 3 ml/min
  - Measuring gas flow for swab test module (optional): 400 ± 10 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

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Specification</th>
</tr>
</thead>
<tbody>
<tr>
<td>Connection</td>
<td>230 V AC, optionally 115 V AC, 50/60 Hz</td>
</tr>
<tr>
<td>Protection</td>
<td>230 V: T6,3 A H</td>
</tr>
<tr>
<td></td>
<td>115 V: T6,3 A H</td>
</tr>
<tr>
<td></td>
<td>(Use only original fuses of Analytik Jena AG)</td>
</tr>
<tr>
<td>Typical average power consumption</td>
<td>400 VA</td>
</tr>
<tr>
<td>PC interface</td>
<td>USB 2.0</td>
</tr>
</tbody>
</table>

### Environmental conditions
### Technical data

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Specification</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature during storage</td>
<td>5 – 55 °C</td>
</tr>
<tr>
<td>Temperature during operation</td>
<td>10 – 35 °C</td>
</tr>
<tr>
<td>Humidity during operation</td>
<td>max. 90 % at +30 °C</td>
</tr>
<tr>
<td>Humidity during storage</td>
<td>(10 – 30) % (use desiccant)</td>
</tr>
<tr>
<td>Air pressure</td>
<td>0.7 - 1.06 bar</td>
</tr>
</tbody>
</table>

### Minimum equipment for the control and analysis unit

<table>
<thead>
<tr>
<th>Requirement</th>
<th>Specification</th>
</tr>
</thead>
<tbody>
<tr>
<td>Operating system</td>
<td>Windows 7 Professional or better</td>
</tr>
<tr>
<td>Processor</td>
<td>Intel Core i3 or better</td>
</tr>
<tr>
<td>Working memory</td>
<td>4 GB</td>
</tr>
<tr>
<td>Free hard disk space</td>
<td>40 GB</td>
</tr>
<tr>
<td>Drive</td>
<td>CD-ROM drive (for software installation)</td>
</tr>
<tr>
<td>Monitor resolution</td>
<td>1024 x 768</td>
</tr>
<tr>
<td>Interfaces</td>
<td>USB 2.0</td>
</tr>
</tbody>
</table>
3 Safety instructions

For your own safety and to ensure error-free and safe operation of the analyzer multi N/C pharma HT, 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.

3.1 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 61010-2-010
- 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.
3.2 Symbols and signal words used

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.
If it is not prevented death or most serious injuries (incapacitation) can result.

**WARNING! DANGER OF ELECTRIC SHOCK IF TOUCHED!**

**CAUTION**
Indicates a potentially hazardous situation.
If it is not prevented light or minor injuries and material damage can result.

**CAUTION! HOT SURFACE!**
Touching the hot surface can cause burns.

**IMPORTANT**
Indicates application hints and other especially useful information without any resulting hazardous or damaging situations.

**ENVIRONMENTAL PROTECTION**
Indicates application hints and information to aid the proper disposal and handling of the substances and materials used.

3.3 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:
### 3.4 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.

### 3.5 Requirements for the operating personnel

The analyzer multi N/C pharma HT 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.

For China:

The device contains regulated substances (according to directive SJ / T 11363-2011). Analytik Jena AG 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.
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 following has to be observed:

- 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!

3.6 Safety instructions, transport and installation

The analyzer is always installed by the customer service department of Analytik Jena AG 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 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.
3.7 Safety instructions - operation

3.7.1 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 decommis-sioned 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 N/C pharma HT and the extension modules must be in good working order. Covered vents or ventilation slits etc. may cause the device to break down or may cause damage to it.
- The furnace operates with temperatures of up to 950 °C. The hot components (furnace, condensation coil) must not be touched during or directly after the operation of the analyzer.
- Keep all combustible materials away from the analyzer.

3.7.2 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.

3.7.3 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 discon-nected 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 AG 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.

### 3.7.4 Safety instructions for compressed gas containers and systems

The carrier gas (oxygen) is taken from compressed gas containers or local compressed gas systems. The required purity of the carrier gas must be ensured (see chapter "Technical data" p. 4)!

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.
- Pipes, hoses, screw connections and pressure reducers for oxygen must be kept free from grease.
- All pipes, hoses and screw connections must be checked regularly for leaks and externally visible damage. Leaks and damaged must be repaired without delay.
- 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!

### 3.7.5 Handling of auxiliary and operating materials

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

When handling 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, handling, 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 labeled 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 (H₃PO₄) or hydrochloric acid (HCl) have to be observed!

The catalyst supplied by the manufacturer (platinum catalyst pharma HT or special catalyst multi N/C) must be treated with the usual caution when handling chemicals.

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!

Avoid dust formation and inhalation of dust when handing the quartz glass wool during filling of the combustion tube.

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.

Ensure good room ventilation in working rooms.

3.7.6 Safety instructions - service and repair

The analyzer is usually serviced by the customer service department of Analytik Jena AG 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).
- Service tasks and the replacement of system components (removal of the combustion tube, catalyst replacement) must only be carried out after a sufficiently long cooling down phase.
- 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 AG. 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!
3.8 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.
4 Technical description

4.1 System design

The analyzer multi N/C pharma HT 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 left-hand removable side wall or the top cover.

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

- Components for sample preparation
- Hose system
- Combustion system
- Components for measuring gas drying and cleaning
- Detectors
- Indicator and control elements, connections
- Electronic component
- Accessories

![Front view (doors open)](image)

Fig. 1 Front view (doors open)
4.1.1 Components for sample preparation

Sample feeding in the analyzer N/C pharma HT takes place via flow injection over a syringe pump with 2-port valve. The injection volume is 50 - 3000 μl.

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

The hoses at the 2-port valve are labeled and connected to the following components:

- Hose no. AA with the sample
- Hose no. AB to the change-over valve
4.1.2 Hose system

4.1.2.1 Hose diagram

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

Fig. 4 Hose diagram
4.1.2.2 Connection method

Within the device most gas connections have been implemented via a FAST connector (FAST – Fast, Save, Tight). 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 HT. 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.

Fig. 6 Fingertight screw connection

4.1.2.3 Components for flow adjustment

The flow of the carrier gas is adjusted automatically in the multi N/C pharma HTS and the inlet flow is controlled via an MFC (mass flow controller). At the device outlet the carrier gas flow is measured using an MFM (mass flow meter), 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 NPOC purging flow is adjusted via a needle valve left to the combustion furnace at the gas box. It is only accessible with the left side wall removed. The NPOC purging flow is measured using an MFM and displayed in the control and analysis software in the window System state.
4.1.2.4 Hose pumps

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 located behind the right door next to the halogen trap.

Phosphoric acid pump

The phosphoric acid pump transports the 10% phosphoric acid to the TIC condensate container with the phosphoric acid being permanently vented.
4.1.3 **Combustion system**

The combustion system is on the rear left side wall of the analyzer.

The combustion furnace is a resistance-heated vertical furnace for digestion temperatures up to 950 °C. Optionally a combined combustion furnace for vertical and horizontal operation can be installed (for operation with the swab test module).

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

The furnace head is fitted to the top opening of the combustion tube. At the bottom end the combustion tube is connected to the condensation coil via a fork clamp.
4.1.4 Components for measuring gas drying and cleaning

4.1.4.1 Condensation coil

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

The analyte gas is cooled down quickly in the condensation coil and the water vapor condenses. The measuring gas water mixture is routed to the TIC condensation container via a hose.

![Condensation Coil Diagram]

1. Hose no. 1 to the TIC condensation container
2. Condensation coil

Fig. 11 Condensation coil

4.1.4.2 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 condensation module is located on the left front side. The measuring gas/water mixture is fed via the top left connection via hose no. 1.

Via the top center connection at the glass container 10% phosphoric acid is automatically provided before each measurement.

The measuring gas is dried by freezing in the cooling block. The dry measuring gas is routed via the top connection out of the TIC condensate container. The measuring gas drying is maintenance free.

The condensate or the waste solution of the TIC detection is automatically pumped out via the lower lateral outlet at the glass container.
4.1.4.3 Water traps

The analyzer multi N/C pharma HT contains two water traps for the greatest possible removal of interfering components in the measuring gas and to protect the detector. They are installed in the gas path after the cooling block (5 in Fig. 12). The larger water trap (TC Prefilter) retains aerosol during operation, the smaller water trap (disposable retention filter) retains rising water.

Fig. 13 Water traps
4.1.4.4  Halogen trap

The analyzer multi N/C pharma HT 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. 14  Halogen trap

4.1.5  Detectors

4.1.5.1  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 wavelengths 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 CO₂.

Measurements using the VITA method

The CO₂ 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 CO₂ measurement (e.g. on account of evaporation and condensation processes when metering the liquid samples) CO₂ 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.
4.1.5.2 Chemiluminescence detector CLD (optional)

The extension of the analyzer multi N/C pharma HT with a Chemiluminescence detector enables the TN₉ detection. The CLD must be positioned as an external device adjacent to the multi N/C pharma HT.

The measuring gas formed by the thermal oxidation of the sample is dried and then enters the reaction chamber of the Chemiluminescence detector for the TN₉ detection. There the nitrogen monoxide present in the measuring gas is oxidized with ozone into activated nitrogen dioxide. By emitting light photons (luminescence) the molecules of the nitrogen dioxide return to their original state. The luminescence is detected using a photomultiplier and is proportional to the nitrogen monoxide concentration. It thus permits the detection of the total nitrogen content of the analyzed sample.

A digestion for the TN₉ detection cannot result in a 100% NO recovery. During the cooling and condensation process of the combustion nitrogen oxides can also occur at higher oxidation levels.

4.1.6 Indicator and control elements, connections

LED displays

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

![Fig. 15 LED to indicate readiness for operation](image)

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

1 internal firmware controller
2 device voltage
3 furnace heating
4 furnace comparator
5 broken thermocouple

![Fig. 16 LED strip (right door open)](image)
Main switch, interfaces, gas connections at the back of the equipment

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 HT.

A diagram at the center of the backplate explains the different connections.

![Diagram of connections on the back of the device]

1. Main switch to switch the analyzer on and off "power switch"
2. Holder for mains fuse "FUSE"
3. Mains connection "main plug"
4. Connection "CLD"
5. Carrier gas connection "O₂"
6. Connection of the neutral conductor at the sampler
7. Waste
8. RS 232 interface for the sampler "sampler"
9. RS 232 interface for CLD module "CLD"
10. USB port for PC

Fig. 17 Connections on the rear of the device

4.1.7 Accessories

The following accessories are required for measurements with the analyzer:
- connection cables, connection hoses
- suitable waste container or drainage
- reagent bottle with drip pan for phosphoric acid H₃PO₄, 250 ml

The reagent bottle must be positioned in the drip pan behind the right door. The reagent bottle is labeled with a safety symbol and the name of the content and must be filled by the user with 10% phosphoric acid.
4.1.8 Extension options for the analyzer

4.1.8.1 Sampler

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.

4.1.8.2 Swab test module (optional)

The combustion system of the multi N/C pharma HT can be extended by a special reactor and a lock with manual feed for the analysis of small quantities of solid samples. Digestion temperatures of up to 950 °C are achieved. The digestion is supported by catalysts.

![Swab test module](image)

Fig. 18 Swab test module

4.1.8.3 Chemiluminescence detector CLD (optional)

For TN₅ detection a Chemiluminescence detector (CLD) can be connected to the analyzer multi N/C pharma HT (see section "Chemiluminescence detector CLD (optional)" p. 23).
### 4.2 Principle of operation

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

![Diagram of Principle of operation](image)

The digestion is performed in the multi N/C pharma HT by thermocatalytic high-temperature oxidation in the presence of special catalysts. This enables a quantitative digestion even for very stable, complex carbon and nitrogen compounds.

The sample aliquot is directly dosed into the hot zone of the filled reactor (combustion tube). Here the pyrolysis and oxidation of the sample in the carrier gas flow is performed with the aid of the catalyst (e.g. equations (1) - (3)). The carrier gas is also used as an oxidation agent.

\[
R + O_2 \rightarrow CO_2 + H_2O \quad (1)
\]
\[
R + N+ O_2 \rightarrow NO + CO_2 + H_2O \quad (2)
\]
\[
R + Cl+ O_2 \rightarrow HCl + CO_2 H_2O \quad (3)
\]

R … carbonic substance

The measuring gas is cooled in the condensation coil and condensed water is separated from the measuring gas in the subsequent TIC condensate container. After further drying and removal of corrosive acting gases, the measuring gas CO₂ is added to the NDIR detector or NO detector (CLD).
Inorganic carbon is detected by injecting a sample aliquot into the acidic TIC reactor and driving out the formed CO$_2$ via the NDIR detector.

The CO$_2$ or NO concentration is detected several times every second. An integer over time is calculated from this signal sequence. The integer is proportional to the concentration of the carbon or nitrogen in the measurement solution. Afterwards, the calculation of the carbon or nitrogen content in the sample is performed via a previously determined calibration function.

### 4.3 Measuring method

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

- **TC** - Total Carbon
- **TOC** - Total Organic Carbon
- **TIC** - Total Inorganic Carbon
- **NPOC** - Non-Purgeable Organic Carbon
- **TN$_b$** - total bound nitrogen (total nitrogen)

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

#### 4.3.1 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 into the combustion tube, digested and the generated carbon dioxide is detected.

Parallel to the TC detection the TN$_b$ detection is possible.

#### 4.3.2 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 (4).

\[
TOC = TC - TIC
\]  
\[\text{(4)}\]

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.

Parallel to the TOC detection the TNb detection is possible.

4.3.3 TIC analysis

During the TIC analysis the total inorganic carbon from carbonates and hydrocarbonates as well as free CO₂ 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 CO₂ is purged and detected.

4.3.4 NPOC analysis

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

The sample is acidified outside of the analyzer with 2 N HCl (pH 2) and the resulting CO₂ is purged. Afterwards the remaining carbon from the sample prepared in this manner is determined via combustion.

Parallel to the NPOC detection the TNb detection is possible.

Other highly volatile organic compounds are purged with the CO₂. The NPOC analysis should not be used when the sample contains easy to purge organic substances.

4.3.5 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 CO₂. 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 CO₂.

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 with 2 N HCl (pH 2) 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.

Parallel to the NPOC plus detection the TNb detection is possible.

Highly volatile organic substances are also purged during the first step and not detected.
4.3.6 **TN₉-analysis**

Parallel to all analyses via high temperature incineration, the detection of the total bound nitrogen is possible in the TN₉ analysis. The thermocatalytic oxidation results in nitrogen oxides which can be detected alternatively using an external Chemiluminescence detector (CLD).

4.4 **Catalysts**

Various suitable solids can be used as catalysts or oxygen carriers in the multi N/C pharma HT supporting the combustion of the components of the sample material in a temperature range of 700 °C to 950 °C.

For the multi N/C pharma HT the use of a special platinum catalyst (included in the scope of delivery of the consumables) with a reaction temperature of 800 °C is recommended. This catalyst has been specially developed and can be used universally over the whole operating range of the analyzer both for the carbon and the nitrogen detection. Because of its very low individual blank value it enables a safe and precise analysis for low carbon and nitrogen contents on the one hand. On the other hand it also works stable during the analysis of highly loaded aqueous materials.

4.5 **Calibration**

4.5.1 **Calibration strategies**

4.5.1.1 **Single point calibration**

For most applications a single point calibration is permitted for the multi N/C pharma HT – 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.

4.5.1.2 **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 2 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.

A standard solution for the range to be calibrated is prepared measured for different volumes in accordance with the settings in the selected method.

### 4.5.1.3 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.

### 4.5.2 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 (5).

\[
F = \frac{c_{\text{nominal}}}{c_{\text{actual}}} \quad (5)
\]

### 4.5.3 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 (6) and (7) through regression calculation.

\[
c = \frac{k_1 \times l_{\text{net}} + k_0}{V} \quad (6)
\]

\[
c = \frac{k_2 \times l_{\text{net}}^2 + k_1 \times l_{\text{net}} + k_0}{V} \quad (7)
\]

- \(c\) … target concentration of the standards
- \(V\) … sample volumes
- \(l_{\text{net}}\) … net integer
- \(k_0, k_1, k_2\) … calibration coefficients
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 or from all individual detections.

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

### 4.5.3.1 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 (6) or (7); the following applies:

\[ c_{TC} = f(I_{TC}) \]  

(8)

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.

### 4.5.3.2 TIC

The TIC channel is calibrated. The calibration functions are calculated in accordance with the equations (6) or (7); the following applies:

\[ c_{TIC} = f(I_{TIC}) \]  

(9)

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.

### 4.5.3.3 TOC (Diff)

Generally separate calibration functions are calculated for the channels TC and TIC in accordance with the equations (6) or (7). The equations (8) and (9) 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 (10).

\[ c_{TOC} = c_{TC} - c_{TIC} \]  

(10)

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.

### 4.5.3.4 NPOC plus

The method NPOC plus is calibrated the same way as the method TOC(Diff). Before the analysis the TIC must be sufficiently purged for the use of the differential method to be meaningful.

- 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 (9) from the calculated concentration difference

**IMPORTANT**

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.

### 4.5.3.5 Calibration TN<sub>b</sub>

The TN channel is calibrated. The calculated calibration functions correspond to the equations (6) or (7); the following applies:

\[ c_{TN} = f(I_{TN}) \]  

(11)

The calculated parameters appear in the method in the TN analysis channel.

### 4.5.4 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.

Verification limit
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.

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

4.5.5 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 are 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.

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

Mean value

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

4.6 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 p-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:

\[ E = \frac{r_{ss} - r_W}{r_S - r_W} \times 100 \] (12)

- \( E \) ... System efficiency 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 %.

4.7 Blank Values

4.7.1 Blank water values

4.7.1.1 Blank preparation water value

Especially for measurements with low TOC concentrations (µg/l range) the TOC content of the water used for preparing the standard can often not be neglected. The weighted-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).

\[ I_{\text{net}} = I_{\text{gross}} - I_{\text{PreparationWater}} \] (13)

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

4.7.1.2 Dilution blank value

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 and entered in 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 dilution blank value is always specified in multiWin standardized to 1 ml.

Use of the dilution blank value:

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 (14)) and deducted from the experimentally determined raw integer (equation (15)). The raw integer determined for each measurement \( I_{raw} \) is corrected by the blank value of the dilution water used.
\[ I_{DBV} = V_{DBV} \times \left( V_{Sample} - \frac{\text{NumberUnitsPrimarySample}}{\text{NumberUnitsDilution}} \times V_{Sample} \right) \] (14)

\[ I_{eff} = I_{raw} - I_{DBV} \] (15)

\[ I_{DBV} \ldots \text{ Dilution blank value} \]
\[ V_{sample} \ldots \text{ sample volumes} \]
\[ I_{eff} \ldots \text{ Effective integer} \]
\[ I_{raw} \ldots \text{ raw integer} \]
\[ I_{DBV} \ldots \text{ Dilution water integer} \]

**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 (16)).

\[ c = \frac{m}{V_{sample}} \times \frac{\text{NumberUnitsDilution}}{\text{NumberUnitsPrimarySample}} \] (16)

For the linear calibration function (equation (6)) the result is then equation (17).

\[ c = k_3 \times I_{eff} + k_0 \times \frac{\text{NumberUnitsDilution}}{V_{sample} \times \text{NumberUnitsPrimarySample}} \] (17)

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.

**4.7.1.3 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 (18)).
\[ I_{\text{eff}} = I_{\text{raw}} - I_{\text{EBV}} \]  \hfill (18)

- \( I_{\text{eff}} \) … Effective integer
- \( I_{\text{raw}} \) … raw integer
- \( I_{\text{EBV}} \) … Eluate blank value

### 4.7.2 Boat blank value

The boat blank value is determined by introducing an empty boat or a boat with additives for the sample into the combustion furnace and analyzing it.

The boat blank value 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.
5 First commissioning

5.1 Site requirements

5.1.1 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 on a heat-resistant and acid-resistant 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!

5.1.2 Space requirement

The AS vario autosampler and the EPA sampler must be positioned on the right of the analyzer. The chemiluminescence detector (CLD) must also be located to the right 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.

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.
5.1.3 Energy supply

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

The analyzer multi N/C pharma HT 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.

5.1.4 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. The length is 3 m. If other lengths are preferred, please contact the customer service department at Analytik Jena AG.

5.2 Unpacking and placing the analyzer

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

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.

**IMPORTANT**
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 HT 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.
6 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 HT when it is switched off!

6.1 Sampler

6.1.1 AS vario autosampler

DANGER OF ELECTRIC SHOCK IF TOUCHED!
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.)!

CAUTION!
Before commissioning the autosampler remove the transport lock. Do not obstruct the sampler during running operation. In both cases 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

<table>
<thead>
<tr>
<th>Max. no. of samples</th>
<th>20</th>
<th>52</th>
<th>72</th>
<th>100</th>
<th>146</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample tubes</td>
<td>100 ml</td>
<td>100 ml</td>
<td>40 and 50 ml</td>
<td>20 ml</td>
<td>12 ml</td>
</tr>
</tbody>
</table>

Technical data

<table>
<thead>
<tr>
<th>Operating voltage</th>
<th>24 V DC via external power supply</th>
</tr>
</thead>
<tbody>
<tr>
<td>Power consumption</td>
<td>50 W</td>
</tr>
<tr>
<td>Grid voltage of external power supply</td>
<td>100 – 240 V, 50 – 60 Hz auto-sensing</td>
</tr>
<tr>
<td>Dimensions (WxDxH)</td>
<td>350 mm x 400 mm x 470 mm</td>
</tr>
</tbody>
</table>

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

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. 20 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 Transport lock
2 Screw M3x12

Fig. 21 Transport lock on autosampler AS vario
1. Place the autosampler on the side as shown in Fig. 21.
2. Unscrew the screw (2 in Fig. 21) 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.

**Commissioning the autosampler**

1. Switch off the analyzer!
2. 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.
3. Plug the grounding conductor into the connection on the rear of the analyzer.
4. 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).
5. 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. **Caution:** Position the outlet tube at a constant incline. If necessary shorten the tube. Tube must not dip in the liquid.
6. Place the sample tray onto the autosampler housing. Make sure it clicks into place.
7. 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.
8. 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. 20)
   - For NPOC measurements with non-parallel purging: Insert both cannulas in one sleeve with two holes in the position on the right (see Fig. 22)

![Sleeve with 2 cannulas for NPOC measurements with non-parallel purging](image)

9. Adjust the cannulas so that approx. one third of the cannulas is visible above the sleeve. Secure the cannulas by slightly tightening the screw.
10. Connect the two connection hoses to the analyzer to the cannulas:
    Hose no. AA = sample aspiration hose
    Hose no. 7 = 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.
11. Attach the sample cover (if present) so that it is positioned in the guide rail.

12. Connect the power supply to the grid.

13. 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. 79).

### 6.1.2 AS 21 autosampler

**DANGER OF ELECTRIC SHOCK IF TOUCHED!**

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.)!

**CAUTION!**

Only adjust the sampler in the switched-off state. Do not obstruct the sampler during running operation. The drives might be damaged.

**Technical data**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Specification</th>
</tr>
</thead>
<tbody>
<tr>
<td>Number of samples</td>
<td>max. 21</td>
</tr>
<tr>
<td>Sample cups</td>
<td>50 ml</td>
</tr>
<tr>
<td>Operating voltage</td>
<td>24 V DC via external power supply</td>
</tr>
</tbody>
</table>
Connecting add-on devices

<table>
<thead>
<tr>
<th>Specification</th>
<th>Detail</th>
</tr>
</thead>
<tbody>
<tr>
<td>Power consumption</td>
<td>30 W</td>
</tr>
<tr>
<td>Dimensions (WxDxH)</td>
<td>260 mm x 350 mm x 310 mm</td>
</tr>
<tr>
<td>Mains voltage of external power supply</td>
<td>100 – 240 V, 50 – 60 Hz auto-sensing</td>
</tr>
</tbody>
</table>

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**

![Layout of the sampler AS 21](image)

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

hose no. AA sample intake hose
Hose no. 7 purging cannula

**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 (6 in Fig. 17 p. 24).

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 (8 in Fig. 17 p. 24).

6. Insert the cannulas and the cannula holder and attach them only lightly using the knurled head screws.

7. Place two sample cups into positions 1 and 2 of the sample tray under the two cannulas.

8. Move the sampler arm down.

9. Adjust the cannulas in height until the cannula tip is 1 to 2 mm away from the bottom of the cup and does not touch the bottom or as required (if using stirrers).

10. Secure the cannulas by tightening the screws lightly.

   **Important:** The screws must not bend the cannulas under any circumstances.

11. Connect the switching power supply to the mains. Switch on the switching power supply.

12. 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]**.

**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.
1. Screw the cannula holder (7) to the autosampler arm.

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. Move the sampler arm down.

6. Adjust the cannulas in height until the cannula tip is 1 to 2 mm away from the bottom of the cup and does not touch the bottom or as required (if using stirrers).

7. Secure the cannulas by tightening the screws lightly.
   **Caution:** The screws must not bend the cannulas under any circumstances.

8. 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.
   **Caution:** The hoses may not hinder the movement of the autosampler arm.

### 6.1.3 AS 10 autosampler

**DANGER OF ELECTRIC SHOCK IF TOUCHED!**

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.)!

**CAUTION!**

Do not obstruct the sampler during running operation. The drives might be damaged.
Connecting add-on devices

Technical data

<table>
<thead>
<tr>
<th>Specification</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Number of samples</td>
<td>max. 10</td>
</tr>
<tr>
<td>Sample tubes</td>
<td>50 ml</td>
</tr>
<tr>
<td>Operating voltage</td>
<td>24 V DC via external power supply</td>
</tr>
<tr>
<td>Power consumption</td>
<td>30 W</td>
</tr>
<tr>
<td>Grid voltage of external power supply</td>
<td>100 – 240 V, 50 – 60 Hz auto-sensing</td>
</tr>
<tr>
<td>Dimensions (Wx Dx H)</td>
<td>160 mm x 130 mm x 300 mm</td>
</tr>
</tbody>
</table>

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

Layout

![Layout of the AS 10 autosampler](image)

Fig. 26 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.


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.

6.1.4 EPA sampler

DANGER OF ELECTRIC SHOCK IF TOUCHED!
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.

CAUTION!
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**

<table>
<thead>
<tr>
<th>Specification</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Number of samples</td>
<td>max. 64</td>
</tr>
<tr>
<td>Sample tubes</td>
<td>40 ml</td>
</tr>
<tr>
<td>Operating voltage</td>
<td>24 V DC via external power supply</td>
</tr>
<tr>
<td>Power consumption</td>
<td>30 W</td>
</tr>
<tr>
<td>Grid voltage of external power supply</td>
<td>100 – 240 V, 50 – 60 Hz auto-sensing</td>
</tr>
<tr>
<td>Dimensions (WxDxH)</td>
<td>500 mm x 550 mm x 470 mm</td>
</tr>
</tbody>
</table>

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

**Layout**

![Diagram of EPA sampler](image)

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

Fig. 27 EPA sampler
Connecting add-on devices

**Fig. 28** 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. 29** 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.).

**Fig. 30** Transport lock

1. Autosampler arm
2. Transport retaining clip
3. Screws
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.

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.
Connecting add-on devices

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. AA = sample aspiration hose
   Hose no. 7 = 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.

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.

Fig. 32 Hose in Fingertight connection

1 Conical nipple
2 Banjo bolt
3 Hose
Connecting add-on devices

- 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. 82).

### 6.2 Chemiluminescence detector (CLD)

**CAUTION**

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

![CLD – display elements, mains connection and media connections](image)

1. LED in the front panel
2. Mains switch
3. Fuse holder
4. Mains connection
5. Serial connection to the analyzer
6. Service interface
7. Connection O₂, synthetic air
8. Connection to the analyzer
9. Gas outlet
10. Adsorption tube (for the removal of NO)

Fig. 33  CLD – display elements, mains connection and media connections
Technical data

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Detection principle</td>
<td>Chemiluminescence detector</td>
</tr>
<tr>
<td>Parameter</td>
<td>TNₐ (Total bound nitrogen)</td>
</tr>
<tr>
<td>Measuring range</td>
<td>0 – 200 mg/l TNₐ</td>
</tr>
<tr>
<td>Detection limit</td>
<td>0.05 mg/l TNₐ</td>
</tr>
<tr>
<td>Analysis time</td>
<td>3 – 5 min</td>
</tr>
<tr>
<td>Gas for ozone generation</td>
<td>Oxygen (recommended) or synthetic air, 60 ml/min, 4 – 6 bar</td>
</tr>
<tr>
<td>Dimensions W x H x D</td>
<td>ca. 300 mm x 460 mm x 550 mm</td>
</tr>
<tr>
<td>Weight</td>
<td>approx. 12 kg</td>
</tr>
<tr>
<td>Connection</td>
<td>110 – 240 V AC, 50/60 Hz</td>
</tr>
<tr>
<td>Protection</td>
<td>2 x T4.0 AH</td>
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<tr>
<td>Typical average power consumption</td>
<td>200 VA</td>
</tr>
<tr>
<td>PC interface</td>
<td>RS 232</td>
</tr>
<tr>
<td>Interference suppression</td>
<td>in accordance with the rules of EN 55011 Group 1, Class B interference-protected (according to EN 61326-1 suitable for use in basic electromagnetic environments)</td>
</tr>
</tbody>
</table>

CAUTION!

The ozone gas (O₃) produced from dry carrier gas in the ozone generator is destroyed in the downstream ozone destroyer if the analyzer is used as intended. In addition, the potential concentration is harmless: Various safety measures result in the automatic shut-down of the ozone generator.

If a smell of ozone occurs in the CLD, switch off the device immediately and contact the Service of Analytik Jena AG.

Connect the Chemiluminescence detector to the analyzer as follows:

1. Place the Chemiluminescence detector to the right of the analyzer.
2. Connect the carrier gas to the connection with the quick-release coupling (6 in Fig. 33 p. 53).
3. Establish the gas connection between CLD and multi N/C pharma HT:
   - CLD connection 7 in Fig. 33 p. 53
   - Analyzer connection 4 in Fig. 17 p.24.
4. Connect the corresponding serial interface “CLD” on the equipment backplate of the analyzer (9 in Fig. 17) via the serial data cable supplied to the RS 232 interface of the Chemiluminescence detector.
5. Switch on the CLD.
   ✓ The lamp on the front panel of the CLD indicates readiness for operation.
6. 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 **Detector** list select the option **CLD**.
− 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 **Sensors** group, activate N-measurement. Exit the **Options** window with [OK].

### 6.3 Swab test module

**CAUTION**

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

The combustion system of the multi N/C pharma HT can be extended by an integrated swab test module for analyzing small quantities of solid samples (swabs). Digestion temperatures of up to 950 °C are achieved. The digestion is performed by catalysts.

#### 6.3.1 Technical data

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Specification</th>
</tr>
</thead>
<tbody>
<tr>
<td>Digestion temperature</td>
<td>up to 950 °C</td>
</tr>
<tr>
<td>Catalyst</td>
<td>CeO₂ (special catalyst for multi N/C)</td>
</tr>
<tr>
<td>Sample volume</td>
<td>max. 500 mg (dependent on the sample material)</td>
</tr>
<tr>
<td>Sample Feed</td>
<td>Boats via manual lock</td>
</tr>
<tr>
<td>Carrier gas supply</td>
<td>Oxygen (min. 4.5)</td>
</tr>
<tr>
<td></td>
<td>preliminary pressure 4 – 6 bar</td>
</tr>
</tbody>
</table>

#### 6.3.2 Structure of the swab test module

The integrated swab test module consists of the following main components:

- Components for sample preparation
- Combustion system
- Accessories

The integrated swab test module is connected via an adapter at the mount of the furnace lock to the analyzer multi N/C pharma HT.

![Fig. 34 Structure of the swab test module](image)

1. Sample feeder
2. Furnace lock with interlock
3. Combustion tube, filled with catalyst
4. Gas outlet adapter
Components for sample preparation

The furnace lock is mounted on the side opening of the combustion tube. In the integrated swab test module the sample feed is via sample boats. The sample volume is max. 500 mg (dependent on the sample material). The sample boat is inserted into the swab test modules via the furnace lock. It is opened and closed manually via an interlock.

Combustion system

The use of the swab test module in the analyzer multi N/C pharma HT requires the use of a combustion furnace for vertical and horizontal operation. The combined combustion furnace permits the operation with a vertically installed combustion tube or the operation with a horizontally installed swab test module.

The horizontally installed combustion tube of the swab test module is made from quartz glass. The furnace lock with manual feed is mounted to the side opening of the combustion tube. The gas transfer hose is connected to the combustion tube adapter and linked to the condensation coil in the analyzer via a fork clamp.

The double-walled combustion tube is filled with catalyst and auxiliary material. As catalyst in the swab test module the special catalyst for multi N/C (CeO₂) with a reaction temperature of up to 950 °C (standard: 900 °C) is used as default.

Accessories

The following accessories are required to operate the swab test module:

- Connection hoses
- Tools

6.3.3 Installing the swab test module

The swab test module can only be installed if a combined combustion furnace for horizontal and vertical operation has been installed.

Filling the combustion tube of the swab test module

CAUTION

Alkaline salts (hand perspiration) causes crystallization in the quartz glass when heating combustion tubes in the combustion furnace. This shortens the service life of the combustion tube.

Where possible do not touch the cleaned combustions tube with your hands for filling. Wear protective gloves during the filling of the combustion tube.

Only fill fully dried combustion tubes. If necessary, dry the combustion tube before filling. Wipe any finger marks on the combustion tube with a cloth wetted with pure alcohol.
1. To fill rotate the adapter (4) of the combustion tube upwards.

2. Insert the quartz wool into the combustion tube, carefully push it down with a glass rod and compact it (2).

3. Carefully fill approx. 60 g CeO₂ (special catalyst multi N/C) via the adapter into the sleeve of the combustion tube (1).

4. Seal the adapter of the combustion tube with some quartz wool (3).

   **Note:**
   The quartz wool is used to retain the catalyst. Seal the adapter to make sure that no catalyst can enter the gas path. However, do not compact the quartz wool too densely.

5. Open the furnace lock:
   - Pull the interlock (1) upwards.
   - Pull the bracket (2) out of its anchorage.
   - Pull the furnace lock open.

6. Release the three hexagon socket screws with the corresponding angular screwdriver by half a turn. Do not fully unscrew the hexagon socket screws.

7. Slide the filled combustion tube up to the stop at the inner ring into the swab test module. The adapter of the gas outlet must then point down.

8. Tighten the hexagon socket screws tightly.

9. Close the furnace lock.
### Installing the swab test module

1. Remove the combustion tube for vertical operation (see section "Removing the combustion tube" p. 87).

2. Remove the sealing plug from the horizontal opening of the combustion furnace and place it onto the vertical opening of the combustion furnace.

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

4. Insert the swab test module into the horizontal opening of the combustion furnace. The adapter of the combustion tube points down.

5. Attach the swab test module with three hexagon socket screws to the holding plate.

6. Attach the gas transfer hose to the adapter of the combustion tube.

7. Combine the gas transfer hose and the inlet of the condensation coil (spherical joint).

8. Secure the spherical joint with the fork clamp and tighten the knurled head screw of the gas clamp finger-tight.
### 6.3.4 Operation of the swab test module

#### Preparing for the measurement

**IMPORTANT**

Oxygen (4.5) must be used as carrier gas when using the swab test module.

Switch on the analyzer as follows:

1. Before switching on the analyzer check that the swab test module has been tightly installed and connected as specified.

2. Switch on the analyzer multi N/C pharma HT.
   
   The carrier gas flow is automatically adjusted to 400 ml/min.

3. Carry out the system leak test (see section "Checking the system for tightness" p. 103).

4. Heat up the combustion furnace. To do so set the furnace temperature in the window *Options / tab Device components* to the desired temperature (e.g. 900 °C).
Tempering the sample boats

**IMPORTANT**

The sample boats might be contaminated. Temper the sample boats prior to analyzing standards and samples. Tempering is performed by an "empty measurement"

After tempering do not touch the sample boat by hand any more. Use e.g. a clean petri dish to store the sample boats and a clean pair of tweezers to transport them.

Note that the swab material may also have a blank value. The swab can be tempered on a boat before wiping. It is also possible to determine the blank value of the swab material and take it into account.

Fold the swab, e.g. using a pair of tweezers, until it can be placed comfortably onto the boat. The height of the folded swab may only exceed the height of the boat slightly

Performing the analysis

*Note:*
When using the swab test module only measurements with manual sample feed are possible.

Prepare the analysis and start the measurement as follows:

1. 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].
   
   *Note:*
   When creating a new method the option **Horizontal furnace** must be selected.

2. In the window **System state** select the manual sample supply by clicking the button [manual].
   
   ✓ This is followed by the initialization of the analyzer.

3. In the window **System state** check the following entries:
   
   − Visual bank - OK
   − Gas flow - OK
   − Temperature - OK
   
   *Note:*
   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. 104.

4. In the window **Options / tab FreeStrings** you may store information about the sample.

5. Click on [Start measurement] or open the menu command **Measurement / Start Measurement**.
   
   The window **Measurement start** opens.

6. Enter the **Sample ID** and, if applicable, a name for the analysis table.

7. Define the **Sample type** for the solid sample to be measured.

8. If applicable, enter explanations about the measurement via the button [Comment].

9. With [Start ▶] open the window **Measurement**.

10. Start the measurement by clicking the button [Start F2] and follow the instructions of the control and analysis software.

11. In the window **Sample** enter the sample volume and then exit with [OK].
12. After a prompt by the information window insert the sample (in the sample boat) into the furnace lock.
   − Open the furnace lock.
   − Insert the sample boats into the furnace lock and hang the eye of the sample boat into the hook of the feeder.

*Note:*
The lock should not be closed, otherwise highly volatile substances are not detected.

![Sample boat inserted into the swab test module](image)

13. Exit the information window by clicking on the button [OK].

14. Follow the prompts of another information window:
   − Close the lock.
   − Slide the sample to the combustion furnace.

*Note:*
If a multiple detection has been agreed in the method, the process restarts at item (10).

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

### 6.3.5 Maintenance of the swab test module

<table>
<thead>
<tr>
<th>Combustion tube</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Maintenance task</strong></td>
</tr>
<tr>
<td>Check for cracks and damages</td>
</tr>
<tr>
<td>Check catalyst and replace if necessary</td>
</tr>
<tr>
<td>Clean combustion tube</td>
</tr>
</tbody>
</table>
Connecting add-on devices

<table>
<thead>
<tr>
<th>Furnace lock</th>
<th>Maintenance task</th>
<th>Maintenance interval</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Check for leaks</td>
<td>3 months</td>
</tr>
<tr>
<td></td>
<td>Replace the sealing ring of the lock</td>
<td>as required</td>
</tr>
</tbody>
</table>

**6.3.6 Removal of the swab test module**

**CAUTION**

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

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

1. In the window *Options / tab Device components* set a furnace temperature of 20 °C.
2. Exit the software multiWin.
3. Switch the analyzer off on the main switch, pull the mains plug from the mains outlet and disconnect the gas supply.
4. Disconnect the plug-in connector on the left-hand side of the swab test module.
5. Pull the gas inlet hose out of the FAST connector at the carrier gas outlet of the gas box. Remove the angled FAST connector from the gas box connection and attach the straight FAST connector.
6. Unscrew the other end of the hose from the swab test module.
7. Remove the fork clamp at the spherical joint between the gas transfer hose and the condensation coil inlet.

8. Release the four knurled head screws at the holding plate and pull the swab test module out of the opening of the combustion furnace.

*Note:* The gas transfer hose and holding plate may remain connected to the swab test module. This makes the next installation of the swab test module easier.

*Caution:* Do not unscrew the angle profiles from the furnace! They are preadjusted and guarantee the correct position of the swab test module during installation.

9. Remove the sealing plug from the vertical opening of the combustion furnace and place it onto the horizontal opening of the combustion furnace.

10. Install the combustion tube for vertical operation (see section "Inserting the combustion tube" p. 90).
7 Operation

7.1 General information for the analysis work

Observe the following during analysis:

- To acidify samples only use hydrochloric acid (HCl) p. A. c = 2 mol/l, made from HCl p. A. (conc.) and TOC water.
- For the TIC determination only 10 % orthophosphoric acid (H₃PO₄), created from orthophosphoric acid (concentrated) p. a. and TOC water are to 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 (CO₂, 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 (difference mode).
  - Manually aerate the head room above the fluids. To do so connect the aeration hose to the gas connection "CLD" and move the other end into the head room of the sample (do not immerse the hose into the fluid!).
  - Remove the source of organic vapors.

7.2 Switching on the analyzer

CAUTION

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.
- Sufficient phosphoric acid is available in the reagent bottle (0.5 ml for each TIC 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.

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

- Sampler
- Chemiluminescence detector (CLD)
- Swab test module
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 additional components (see user manual of the respective component):
   - Sampler
   - Chemiluminescence detector (CLD)
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.

**IMPORTANT**

In the window **System state** the displays of the components which are not yet ready are shown in red during initialization. During the start-up phase of the analyzer the external communication with the program is blocked.

The individual components have different start-up periods:

- NDIR detector approx. 10 minutes start-up period
- Furnace approx. 10 minutes heating-up time
- CLD approx. 30 minutes running-in time

The measuring gas flow reaches the target value (120 ± 10 ml/min) after approx. 1 to 2 minutes.

7. If the analyzer is not ready for measurements after 35 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. 104.

8. If necessary, adjust the NPOC purging flow (see section "Adjust the NPOC purge flow" p. 81).

**Note:**
The NPOC purging flow is set to approx. 100 ml/min and can be increased or reduced dependent on the measuring task.
7.3 Carrying out the calibration

7.3.1 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 of new calibration** is opened.

4. In the group **Calibration parameters** select the calibration type.

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

   For multipoint calibrations with variable sample volumes and constant concentrations the option **constant sample volumes** must be enabled. The corresponding 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.
   
   **Note:**
   Not all parameters need necessarily be calibrated. For the calibration of the parameter NPOC plus and concentrations > 0.5 mg/l the parameters IC and TC must be activated individually.

   The calibration for the parameter TOC/NPOC plus must be used when working in a concentration range < 0.5 mg/l. Here a single point calibration is generally sufficient.

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 black** 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.

   **Note:**
   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.

   **Note:**
   The number of repeat measurements configured in the method is entered automatically in the column **Rep.**. 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...**.

    **Note:**
    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.

    **Note:**
    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).
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.

### 7.3.2 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 via 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.
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 is based on individual values or average values of the net integer. For the selected regression type the respective calibration coefficients are displayed.

Calibration graph
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].

Fig. 38 Calibration window - Data of new calibration
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.32.

### 7.3.3 Editing an existing calibration

**IMPORTANT**
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. 38). 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. 39). Individual values can be disabled by removing the (✓) in the column **use**.
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].

  **Note:**
  Measuring points can only be added individually.

### 7.3.4 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).

   **Note:**
   Up to three linear calibration ranges for each parameter can be stored in a method. It should be ensured that the ranges merge and do not have any gaps! 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 (✓).

   **Note:**
   Not all calibrated parameters need to be transferred to the method.

3. Click on the [Link to Method] button.
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 the selected method

*Note:*
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: xxx** 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.

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

![Fig. 40 Window Link to Method](image)

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

<table>
<thead>
<tr>
<th>No calibration range exists</th>
<th>- Transfer the currently determined calibration data with the button [Accept values]. The same calibration coefficients appear in the left-hand and right-hand columns.</th>
</tr>
</thead>
</table>
| 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.  
- 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.
- **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. 41, table, first line)
- The accepted calibration parameters are used for the calculation of all subsequent analyses with this method.

---

**Fig. 41**  
*Window Link with method with three areas*

### 7.3.5 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.

**Note:**
In the window **Selection CalibrationReport** filters can be set, if necessary, and the records sorted by clicking on the respective header.

3. Highlight the corresponding calibration report and click on the button **[OK]**.

   ✓ The calibration report is displayed.
7.4 Performing measurements

7.4.1 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
   − where applicable, CLD - OK
   − Gas flow - OK
   − Temperature - OK
   Note:
   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. 104.
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.

7.4.2 Measurement with sampler

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

1. 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].
2. In the window **System state** select sample supply with sampler by clicking the button [Sampler].
   ✓ **This is followed by the initialization of the analyzer.**

3. In the window **System state** check the following entries:
   - Visual bank - OK
   - if applicable CLD - OK
   - Gas flow - OK
   - Temperature - OK

   **Note:**
   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. 104.

4. Fill the sample cups with the measuring liquid and place them onto the sample tray.

5. 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.**
# 8 Maintenance and care

## 8.1 Maintenance intervals

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<td>daily</td>
</tr>
<tr>
<td></td>
<td>Exchange water trap</td>
<td>as required, but no later than after 6 months</td>
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<th>Maintenance task</th>
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<td>Check copper wool for discoloring</td>
<td>daily</td>
</tr>
<tr>
<td></td>
<td>Replace depleted copper/brass wool</td>
<td>if half of the copper wool or the brass wool is discolored</td>
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<table>
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<th>Maintenance interval</th>
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</tr>
<tr>
<td></td>
<td>Check catalyst and replace if necessary</td>
<td>as required, after corresponding message in multiWin at the latest</td>
</tr>
<tr>
<td></td>
<td>Clean combustion tube</td>
<td>during catalyst replacement</td>
</tr>
<tr>
<td></td>
<td>Renew (replace) combustion tube</td>
<td>as required, but no later than after 12 months</td>
</tr>
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<table>
<thead>
<tr>
<th>TIC condensate container</th>
<th>Maintenance task</th>
<th>Maintenance interval</th>
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<td>Check for cracks and damages</td>
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</tr>
<tr>
<td></td>
<td>Clean the condensation coil</td>
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<th>Maintenance task</th>
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<tr>
<td></td>
<td>Check for leaks</td>
<td>3 months</td>
</tr>
<tr>
<td></td>
<td>Replace porous pump hose</td>
<td>as required, but no later than after 12 months</td>
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<th>Maintenance task</th>
<th>Maintenance interval</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Check for leaks</td>
<td>3 months</td>
</tr>
<tr>
<td></td>
<td>Replace porous pump hose</td>
<td>as required, but no later than after 12 months</td>
</tr>
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<table>
<thead>
<tr>
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<th>Maintenance task</th>
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</thead>
<tbody>
<tr>
<td></td>
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<td>3 months</td>
</tr>
<tr>
<td></td>
<td>Clean the dosing syringe</td>
<td>as required, but no later than after 12 months</td>
</tr>
</tbody>
</table>

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

**IMPORTANT**
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. 103).
8.2 Adjustment and setup tasks

8.2.1 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 "Sampler" section, p. 40.

8.2.2 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!

**IMPORTANT**

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 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.

![Adjustment points on the sample tray](image)

**Fig. 43** 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.

![Screenshot of the multiWin® software](image)

7. To adjust position 1, place a sample tube with magnetic stirrer onto the sample tray.
   
   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).

8. 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.
9. Click [Save].
   ✔ The adjustment values will be taken over.

10. Open the Alignment sampler window again and click the corresponding button to move to the rinse position/position 1 again to check the alignment.

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

8.2.3 Adjust the NPOC purge flow

CAUTION
There is a risk of burning on the combustion furnace! Proceed with the greatest care when adjusting the NPOC purging flow via the NPOC needle valve!

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 NPOC needle valve is located behind the left side wall adjacent to the combustion furnace.

Adjust the NPOC purge flow as follows:

1. Open the left side wall of the analyzer. Unscrew the four fastening screws; the screws are undetachable and remain in the wall. Disconnect the grounding conductor and safely put the side wall aside.

2. In multiWin use the menu command Instrument / Device control to open the window Device control.

3. From the list field select the option Purging.

4. 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.
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For manual sample supply:

- Select the purging time in the field **Time** between 1 and 900 seconds.
- Insert the purging hose into the cup filled with ultrapure water for which the purging flow is to be adjusted.

5. Click on the button *[Start F2]*.

6. Undo the adjustment screw at the NPOC needle valve.

7. Regulate the desired NPOC purge flow:
   - Increasing the NPOC purging flow - turn needle valve to the left
   - Reducing the NPOC purging flow - turn needle valve to the right
   - In the window **System state** check the flow indication

8. Relock the adjustment screw at the needle valve.

9. Close the side wall.
   Connect the grounding conductor connection to the left side wall.
   First screw the screws into the bottom and then the top side. Tighten the screws in turn.

### 8.2.4 Adjusting the EPA sampler

**CAUTION**

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

**IMPORTANT**

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

**IMPORTANT**

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 but does not enter the sample liquid when delivering the acid.

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

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

   **Caution:**
   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.

   **Note:**
   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.

   **Note**
   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.

   **Note:**
   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.
8.3 Replacing the water traps

**IMPORTANT**
The water traps (TC Pre-filter and disposable retention filter) can be replaced in the switched-on state but not during a measurement. Always replace both water traps!
The water traps only serve their function if they are inserted in the order and installation direction specified!

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

1. Open the doors of the analyzer.
2. Undo the connection (1) to the halogen trap with a single turn.
3. Pull the water trap out of the hose (5) at the TiC container.
4. Assemble the new water traps.
   
   **Note:**
   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. Attach the large water trap to the hose at the TiC container (5).
6. Press the water traps into the clamp (4) on the equipment backplate
7. Screw hose no. 2 to the halogen trap at the adapter of the small water trap finger-tight.
8. Check the system for leaks (see section "Checking the system for tightness" p. 103).
9. Close the front doors.
8.4 Replacing the halogen trap

**CAUTION**
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 is discolored black 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. Open the doors of the analyzer.
2. Pull the FAST connectors (1 & 2) off the halogen trap and pull the U tube out of the clamps (3).
3. Pull out the depleted copper or brass wool from the U-tube with a tweezers or a small hook.
4. Check the U-tube for cracks.
   **Note:**
   Only reuse a fully intact U tube!
5. If required, flush the U-tube with ultrapure water and leave to dry completely.
6. Fill the U-tube with new copper and brass wool using tweezers or a small hook.
   **Note:**
   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.
7. Cover the copper and brass wool with cotton wool.
8. Press the filled U tube carefully into the clamps.
9. Connect hose no. 2 to the gas inlet branch with copper wool and hose no. 3 to the gas outlet branch with brass wool.
10. Check the system for leaks (see section "Checking the system for tightness" p. 103).
11. Close the doors of the analyzer.
### 8.5 Exchanging the catalyst

#### 8.5.1 Lifetime of the catalyst

If the effectiveness of the catalyst decreases, the combustion tube has to be filled again. A check has to be performed after expiry of the service interval (maximum 1500 injections). The expiry of the maintenance interval is displayed in multiWin by a message.

#### 8.5.2 Removing the combustion tube

**CAUTION**

There is a risk of burning on the combustion furnace! Only remove the combustion tube when the device is cold or allow the device to cool down sufficiently! Before switching off set the furnace temperature in multiWin to 20 °C and exit multiWin. Otherwise there is a risk of burns when checking the system for leaks after installation!

Remove the combustion tube as follows:

<p>| | |</p>
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<tr>
<th></th>
<th></th>
</tr>
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<tbody>
<tr>
<td>1.</td>
<td>Switch the analyzer off on the main switch, pull the mains plug from the mains outlet and disconnect the gas supply.</td>
</tr>
<tr>
<td>2.</td>
<td>Remove the top cover. Open the left side wall of the analyzer. Unscrew the four fastening screws; the screws are undetachable and remain in the wall. Disconnect the grounding conductor connection and put the side wall safely aside.</td>
</tr>
<tr>
<td>3.</td>
<td>Pull the carrier gas cannula out of the FAST connector at the right side wall.</td>
</tr>
<tr>
<td>4.</td>
<td>Unscrew the Fingertight connection of the furnace cannula from the change-over valve.</td>
</tr>
</tbody>
</table>
5. Undo the knurled head screw at the holder of the change-over valve. Slide the change-over valve to the right. This pulls the furnace cannula out of the change-over valve.

6. Remove the fork clamp below the combustion furnace that connects the outlet of the combustion tube to the condensation coil.

7. Carefully pull the combustion tube upwards out of the combustion furnace.

8. Unscrew the furnace head from the combustion tube and remove the union nut, the pressure ring and the three sealing rings.

9. Remove the used catalyst filling (for notes on disposal see section "Disposal" p. 122).

10. Check the combustion tube for excessive crystallization, cracks and blown out places.

   **Note:** Only reuse intact combustion tubes.

11. If required, flush the empty combustion tube carefully with ultrapure water and leave to dry completely.

### 8.5.3 Filling the combustion tube

**CAUTION! CONTAMINATION AT THE COMBUSTION TUBE!**

Alkaline salts (hand perspiration) causes crystallization in the quartz glass when heating combustion tubes in the combustion furnace. This shortens the service life of the combustion tube. Where possible do not touch the cleaned combustion tube with your hands for filling. Wear protective gloves during the filling of the combustion tube. Only fill fully dried combustion tubes. If necessary, dry the combustion tube before filling. Wipe any finger marks on the combustion tube with a cloth wetted with pure alcohol.
### Filling the combustion tube for conventional samples

You can fixate the combustion tube on a tripod for filling. Fill the combustion tube in accordance with the following instruction from the bottom up:

1. Insert the quartz glass wool approx. 1 cm thick into the combustion tube, carefully push it down with a glass rod and compact it. 
   **Note:** Don’t press too tight! The quartz glass wool is used to retain the catalyst. Make sure that no catalyst can enter the subsequent gas path.

2. Place the platinum catalyst for multi N/C carefully onto the quartz glass wool (approx. 4 cm high).

3. Roll up the high temperature fiber mat (HT mat) of the narrow side. The roll must have a diameter of about 13 mm and a height of 2 cm, so it can easily slide into the combustion tube.

   Insert the rolled HT mat in the combustion tube and slide the HT mat with a glass rod far enough that the catalyst is covered. Press the mat only slightly onto the catalyst.

The recommended work temperature for this filling is 800 °C.

| 1 | Quartz wool, approx. 1 cm |
| 2 | Platinum catalyst, approx. 4 cm |
| 3 | rolled-up HT mat, approx. 2 cm high |

### Filling the combustion tube for samples with high salt concentrations

For samples with high salt concentrations the catalyst is filled on a **platinum net**.

You can fixate the combustion tube on a tripod for filling. Fill the combustion tube in accordance with the following instruction from the bottom up:

1. Position the platinum net in the combustion tube, and carefully push it down with a glass rod. 
   **Note:** The platinum net should retain the catalyst. Make sure that no catalyst can enter the subsequent gas path.

2. Place the platinum catalyst for multi N/C carefully onto the platinum net.

3. Roll up the HT mat from the narrow side. The roll must have a diameter of about 13 mm and a height of 2 cm, so it can easily slide into the combustion tube.

   Insert the rolled HT mat in the combustion tube and slide the HT mat with a glass rod far enough that the catalyst is covered. Press the mat only slightly onto the catalyst.

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

| 1 | Platinum net |
| 2 | Platinum catalyst, approx. 4 cm |
| 3 | rolled-up HT mat, approx. 2 cm high |
8.5.4 Inserting the combustion tube

**CAUTION**
Alkaline salts (hand perspiration) cause crystallizations in the quartz glass when heating the combustion furnace which reduce the service life of the combustion tube. Where possible do not touch the cleaned combustions tube with your hands. Wear protective gloves to install the lock on the combustion tube. If necessary, clean the combustion tube externally before installing it in the combustion furnace (e.g. by wiping it with moist cellulose).

**IMPORTANT**
It is best to fit the furnace head onto the combustion tube before installing the combustion tube in the combustion furnace.

Insert the combustion tube as follows:

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

2. Place the pressure ring (2) into the union nut.
   
   **Note:**
   The conical side of the pressure ring must point up.

3. Slide the three coated sealing rings (3) onto the combustion tube.
   
   **Note:**
   Make sure that the sealing rings at the edge of the combustion tube are flush.

4. Carefully place the furnace head onto the combustion tube up to the stop; press slightly against it and tighten the union nut finger-tight. The furnace head is fully configured with the furnace cannula and the cannula for the carrier gas.
5. Position the ceramic holder in the top opening of the combustion furnace.
6. Insert the combustion tube with the furnace head into the combustion furnace.

7. Attach the bottom end of the combustion tube and the inlet of the condensation coil (spherical joint (1)).
8. Secure the spherical joint with the fork clamp (2) and tighten the knurled head screw (3) finger-tight.

9. Connect the carrier gas connection to the connection at the gas box using the FAST connector.

10. Slide the valve to the left until the valve contacts the connection of the furnace cannula.
11. Screw the furnace cannula with the Finger-tight connection finger-tight to the valve.

12. Secure the valve in this position by tightening the knurled head screw at the holder finger-tight.
13. Replace the top cover.
14. Close the side wall.
   Connect the grounding conductor connection to the left side wall.
   First screw the screws into the bottom and then the top side. Tighten the screws in turn.

5. Switch on the gas supply, insert the mains plug into the power socket and switch on the analyzer on the main switch.
6. Check the system for leaks (see section "Checking the system for tightness" p. 103).

**CAUTION**
The catalyst may emit gas during first heating (mist formation in the TIC condensate container). It must therefore be tempered at operating temperature for approx. 30 min. during first heating (until no more mist forms).
During this time remove the water traps from the TIC container to interrupt the gas path to the detector.

**8.6 Cleaning the TIC condensate vessel**

**WARNING**
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 condensate vessel as follows and clean it:

1. Exit the control and analysis software multiWin.
2. Open the doors of the analyzer.
3. Remove connection hose to the water cascades (1) from TIC condensate container.
4. Remove hose No. 1 AD, BB including the quick release connection from the TIC condensate vessel.
5. Disconnect the waste hose no. 11 to from the bottom connection on the TIC condensate vessel.
6. Undo the 2 knurled head screws (2) at the cover of the cooling block and remove the cover and take out the TIC condensate vessel.
7. Pull the quick-release connections off the connectors of the TIC condensate container.
8. Check the TIC condensate vessel for deposits and cracks.
9. If required, rinse the TIC condensate vessel with ultrapure water.
10. Attach the hoses as shown in the adjacent figure:
   - Insert waste hose No. 11 at least 1 cm into the lower side connection on the TIC condensate vessel.
   - Inserte hose No. 1 AD and BB including quick release connector to the connection of the TIC condensate vessel.

   Note: Quick-release connector needs to be inserted at least 1 cm onto the glass support on the TIC condensate vessel.
   - Push hose no. 1 almost to the bottom of the TIC condensate container.
8.7 Removing and installing the condensation coil

8.7.1 Removing and cleaning the condensation coil

Remove the condensation coil as follows:

1. Switch the analyzer off on the main switch, pull the mains plug from the mains outlet and disconnect the gas supply.

2. Open the left side wall of the analyzer. Unscrew the four fastening screws; the screws are undetachable and remain in the wall. Disconnect the grounding conductor connection and put the side wall safely aside.

3. Pull hose 1 off the FAST connector of the condensation coil.

4. Undo the knurled head screw at the fork clamp and remove the fork clamp connecting the outlet of the combustion tube to the condensation coil.

5. Carefully pull the bottom portion of the condensation coil out of the cut-out in the combustion furnace.

6. Then pull the FAST connector off the glass adapter of the condensation coil.

7. Check the condensation coil for deposits and cracks.

8. If required, flush the condensation coil with ultrapure water and leave to dry completely.
8.7.2 Inserting the condensation coil

Insert the condensation coil as follows:

1. Insert the holder for the condensation coil into the clamps on the right-hand side of the furnace (1).
2. Push the FAST connector onto the adapter of the condensation coil.
3. Push hose 1 into the FAST connector.
4. Place the condensation coil onto the holder. The spherical joint of the coil protrudes into the lower cut-out of the furnace.
5. Attach the bottom end of the combustion tube and the inlet of the condensation coil (spherical joint (1)).
6. Secure the spherical joint with the fork clamp (2) and tighten the knurled head screw (3) finger-tight.
7. Adjust the holder of the condensation coil until the holder supports the glass body well.
8. Close the side wall. Connect the grounding conductor connection to the left side wall. First screw the screws into the bottom and then the top side. Tighten the screws in turn.

9. Switch on the gas supply, insert the mains plug into the power socket and switch on the analyzer on the main switch.

10. Check the system for leaks (see section "Checking the system for tightness" p. 103).

8.8 Cleaning and replacing the metering syringe

Replace or clean the metering syringe 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. Unscrew the metering syringe from the valve (1) and remove it from the drive (3).

4. Dismantle and clean the glass cylinder (2) and piston (4).

5. Insert the piston rod of the new metering syringe into the drive.

6. Screw the glass cylinder to the valve.
8.9 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 and the phosphoric acid pump every 3 months or after every catalyst replacement for leaks.

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 to the left.

3. Pull the hoses no. 10 and no. 11 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.

   Note:
   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.

   Note:
   If the pump body and roller carrier are heavily corroded, please contact Service.

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

   Note:
   During installation the hose clamps must be rotated downwards. Push the hose guide into the groove on the conveyor belt.
9. Position the conveyor belt around the pump body.
10. Press the conveyor belt upwards with one hand and rotate the bracket with the other hand to the right until it engages.
11. Push hose no. 10 and hose no. 11 back onto their adapters.

12. Check the system for leaks (see section "Checking the system for tightness" p. 103).

Phosphoric acid pump

Remove the pump hose analog to the removal for the condensate pump and inspect it for leaks.

The hoses no. AC and AD are connected to the pump using Fingertight connections. Unscrew the connections from the connectors during removal and tighten them again after installing the pump hose.
8.10 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. 103).

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.
8.11 Removing and installing the combustion furnace

8.11.1 Removing the combustion furnace

CAUTION

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

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

1. Switch the analyzer off on the main switch, pull the mains plug from the mains outlet and disconnect the gas supply.
2. Remove the top cover.
3. Open the left side wall of the analyzer. Unscrew the four fastening screws; the screws are undetachable and remain in the wall. Disconnect the grounding conductor connection and put the side wall safely aside.
4. Remove the condensation coil (see section "Removing and cleaning the condensation coil" p. 94).
5. Remove the combustion tube (see section "Removing the combustion tube" p. 87).
   Slide the valve to the right to prevent it from obstructing the removal.
6. Pull the plug-in connector for the combustion furnace out of its socket.
7. Undo the knurled head screws at the retention plates of the furnace at the device floor. Slide the plates to the outside.
8. Lift the combustion furnace out of the analyzer.
### 8.11.2 Installing the combustion furnace

Install the combustion furnace as follows:

<table>
<thead>
<tr>
<th>Step</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Remove the top cover.</td>
</tr>
<tr>
<td>2.</td>
<td>Open the left side wall of the analyzer. Unscrew the four fastening screws; the screws are undetachable and remain in the wall. Disconnect the grounding conductor connection and put the side wall safely aside.</td>
</tr>
<tr>
<td>3.</td>
<td>Undo the knurled head screw at the holder of the valve and slide the valve to the right.</td>
</tr>
<tr>
<td>4.</td>
<td>Undo the knurled head screws at the retention plates on the floor of the analyzer and slide the retention plates outwards.</td>
</tr>
<tr>
<td>5.</td>
<td>Place the furnace centered between the retention plates and align its front parallel to the equipment wall. Slide the retention plates over the furnace feet and tighten the knurled head screws finger-tight.</td>
</tr>
<tr>
<td>6.</td>
<td>Plug the plug-in connector for the combustion furnace into the socket at the bottom right of the rear equipment wall.</td>
</tr>
<tr>
<td>7.</td>
<td>Insert the combustion tube (see section &quot;Inserting the combustion tube&quot; p. 90).</td>
</tr>
<tr>
<td>8.</td>
<td>Install the condensation coil (see section &quot;Removing and cleaning the condensation coil&quot; S. 94).</td>
</tr>
</tbody>
</table>
9. Push the sample intake hose and the purging hose through the top aperture. Attach the cover.

10. Close the side wall.
    Connect the grounding conductor connection to the left side wall.
    First screw the screws into the bottom and then the top side. Tighten the screws in turn.

11. Switch on the gas supply, insert the mains plug into the power socket and switch on the analyzer on the main switch.

12. Check the system for leaks (see section "Checking the system for tightness" p. 103).

8.12 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 HT.
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): 120 ml/min
   - Out (outlet flow): 120 ml/min (± 10 ml/min)

**CAUTION!**

If the outlet flow is significantly below the inlet flow of 120 ml/min, all connection points must be reinspected.
9  

Fault removal

9.1  
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 AG must always be informed.

As soon as the multi N/C pharma HT 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.

For fault analysis it is possible to record log files. The recording of log files should be enabled after consultation with the Analytik Jena AG service department for specific faults. The log files are stored in the directory \multiWin\LOG.

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 line parameter C (see software description "multiWin")

- multiWin_MEM.*:
  - Log file to monitor the RAM memory capacity
  - activation by program start with command line parameter MEM (see software description "multiWin")

- 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 (✓).

IMPORTANT

For fault diagnosis the complete directory \multiWin\LOG must be emailed to the Service department of Analytik Jena AG (see Service address on the inside front cover).

IMPORTANT

If the errors below cannot be remedied using the corresponding fault removal notes, the Service department of Analytik Jena AG must always be informed. This also applies for the repeated occurrence of individual faults.
## 9.2 Error messages in multiWin

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

<table>
<thead>
<tr>
<th></th>
<th>COM 2 not found</th>
<th>Remedy</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>COM 3 not found</td>
<td>switch analyzer off/on</td>
</tr>
<tr>
<td></td>
<td>COM 4 not found</td>
<td></td>
</tr>
</tbody>
</table>

#### Cause
- problems with internal hardware

#### Remedy
- switch analyzer off/on

<table>
<thead>
<tr>
<th></th>
<th>COM 2 not found</th>
<th>Remedy</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>COM 3 not found</td>
<td></td>
</tr>
<tr>
<td></td>
<td>COM 4 not found</td>
<td></td>
</tr>
</tbody>
</table>

#### Gas pressure error

<table>
<thead>
<tr>
<th></th>
<th>Cause</th>
<th>Remedy</th>
</tr>
</thead>
<tbody>
<tr>
<td>10</td>
<td>Counterpressure in the analysis system too great: carrier gas supply is automatically interrupted to protect the analyzer; flow indication MFC approx. 0 ml/min</td>
<td>search for and replace component causing the gas pressure error</td>
</tr>
<tr>
<td></td>
<td>water trap clogged</td>
<td>undo connection upstream of the water traps and reinitialize the analyzer</td>
</tr>
<tr>
<td></td>
<td></td>
<td>check if gas pressure error occurs again, if not, replace water traps</td>
</tr>
<tr>
<td></td>
<td>no gas flow at the measuring outlet (gas supply hose for sample gas supply kinked)</td>
<td>check gas supply hose, remove kink if necessary</td>
</tr>
<tr>
<td></td>
<td>condensation coil clogged with catalyst balls</td>
<td>interrupt measuring gas flow between the combustion tube and condensation coil ( \Rightarrow ) check if &quot;gas pressure error&quot; occurs again, if not - rinse condensation coil clear with ultrapure water</td>
</tr>
<tr>
<td></td>
<td></td>
<td>during catalyst replacement always make sure that sufficient quartz glass wool is filled as a first layer</td>
</tr>
<tr>
<td></td>
<td>combustion tube &quot;solidified&quot; (by analysis of highly saline samples accumulation of salt in the combustion tube)</td>
<td>replace HT mat in the combustion tube or replace catalyst (dependent on the number of measurements with the current catalyst filling and activity of the catalyst)</td>
</tr>
<tr>
<td></td>
<td>HT mat used up by analysis of highly saline samples</td>
<td></td>
</tr>
<tr>
<td></td>
<td>gas supply to the furnace head clogged</td>
<td>clean gas supply to the furnace head</td>
</tr>
</tbody>
</table>

#### Incorrect version number

<table>
<thead>
<tr>
<th></th>
<th>Cause</th>
<th>Remedy</th>
</tr>
</thead>
<tbody>
<tr>
<td>12</td>
<td>the versions of multiWin and the software of the internal computer do not match</td>
<td>update as appropriate</td>
</tr>
</tbody>
</table>

#### No connection to the autosampler

<table>
<thead>
<tr>
<th></th>
<th>Cause</th>
<th>Remedy</th>
</tr>
</thead>
<tbody>
<tr>
<td>13</td>
<td>sample not switched on</td>
<td>switch on sampler and initialize analyzer</td>
</tr>
<tr>
<td></td>
<td>connection cable not connected or faulty</td>
<td>check connection cable</td>
</tr>
</tbody>
</table>

---

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| 20  | no connection to optics (NDIR) | Cause                  | Remedy                        |
| 21  | CRC error optics              | − communication error   | − initialize analyzer         |
| 22  | status error optics           | − NDIR detector faulty  | − inform Service              |
| 26  | optics error; incorrect command return | |

| 24  | Optics error, analog values outside of range | Cause                                      | Remedy                                      |
| 30  | No connection to the N sensor | − the analog values of the detector are outside the working range | − check the quality of the carrier gas (oxygen 4.5 required) |
| 40  | no connection to the syringe pump | − the analog values of the detector are outside the working range | − initialize analyzer and check analog values via component test |

| 111 | Rotator error | Cause | Remedy                                      |
| 112 | Swivel drive error | − Drive incorrectly positioned, e.g. jammed | − Initialize analyzer |
| 113 | Lifting drive error | − Drive incorrectly positioned, e.g. jammed | − If the error cannot be corrected, contact Service |
| 114 | Rack detection error | − Drive faulty | − Initialize analyzer |

| 111 | Rotator error | Cause | Remedy                                      |
| 114 | Rack detection error | − Drive incorrectly positioned, e.g. jammed | − Initialize analyzer |
| 113 | Lifting drive error | − Drive faulty | − If the error cannot be corrected, contact Service |

| 112 | Swivel drive error | Cause | Remedy                                      |
| 113 | Lifting drive error | − Drive correctly positioned | − Initialize analyzer |
| 114 | Rack detection error | − Drive faulty | − If the error cannot be corrected, contact Service |

| 111 | Rotator error | Cause | Remedy                                      |
| 114 | Rack detection error | − Sample tray not positioned correctly | − Position the sample tray again and make sure it clicks into place |
| 113 | Lifting drive error | − Drive faulty | − Initialize analyzer |
| 112 | Swivel drive error | − Drive correctly positioned | − Initialize analyzer |
| 113 | Lifting drive error | − Drive faulty | − If the error cannot be corrected, contact Service |

| 114 | Rack detection error | Cause | Remedy                                      |
| 111 | Rotator error | − Drive incorrectly positioned, e.g. jammed | − Initialize analyzer |
| 112 | Swivel drive error | − Drive faulty | − If the error cannot be corrected, contact Service |
## Fault removal

<table>
<thead>
<tr>
<th>Code</th>
<th>Description</th>
<th>Cause</th>
<th>Remedy</th>
</tr>
</thead>
<tbody>
<tr>
<td>115</td>
<td>Wrong rack</td>
<td>− Wrong sample tray set in the software</td>
<td>− Check settings in the software (see section 6.1); if necessary, set a different sample tray</td>
</tr>
<tr>
<td>116</td>
<td>Unknown sampler command</td>
<td>− Communication error</td>
<td>− Contact Service</td>
</tr>
</tbody>
</table>
| 200  | Restart computer in the analysis device          | − internal computer reset, overvoltage, short-term power failure     | − if the front LED indication (Lockin) illuminates, initialize the analyzer  
|      |                                                 |                                                                       | − for repeated occurrence monitor precisely at which time the error occurs (note status line) |
| 201  | Restart the internal program                     | − internal program error                                              | − initialize analyzer                                                  
|      |                                                 |                                                                       | − for repeated occurrence monitor precisely at which time the error occurs (note status line) |
| 202  | File method.txt not found                        | − program error                                                       | − inform Service                                                        |
| 203  | File init.cnf not found                          |                                                                       |                                                                        |
| 401  | Syringe pump: Initialization                     | − communication error                                                 | − initialize analyzer                                                  |
| 402  | Syringe pump: invalid command                    | − syringe pump faulty                                                 | − inform Service                                                        |
| 403  | Syringe pump: invalid operand                    |                                                                       |                                                                        |
| 404  | Syringe pump: faulty command sequence            |                                                                       |                                                                        |
| 407  | Syringe pump: syringe pump not initialized       | − analyzer not yet initialized after switching on                    | − initialize analyzer                                                  |
|      |                                                 | − reset the syringe pump                                              |                                                                        |
| 409  | Syringe pump: pump sluggish                      | − clogging of a hose line (AA) or (AB)                               | − search for cause and remedy fault                                   
<p>|      |                                                 |                                                                       | − replace hose line - if necessary remove hose line and rinse with ultrapure water, then reinstall |
|      |                                                 |                                                                       | − initialize analyzer                                                  |
|      |                                                 |                                                                       | − inform Service                                                        |</p>
<table>
<thead>
<tr>
<th>Code</th>
<th>Fault Description</th>
<th>Cause</th>
<th>Remedy</th>
</tr>
</thead>
<tbody>
<tr>
<td>410</td>
<td>Syringe pump: valve sluggish</td>
<td>− syringe pump faulty</td>
<td>− inform Service</td>
</tr>
<tr>
<td></td>
<td></td>
<td>− valve broken</td>
<td></td>
</tr>
<tr>
<td>411</td>
<td>Syringe pump: pump step not permitted</td>
<td>− communication error</td>
<td>− initialize analyzer</td>
</tr>
<tr>
<td>415</td>
<td>Syringe pump: Invalid command</td>
<td>− syringe pump faulty</td>
<td>− inform Service</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>MESSx</td>
<td>Analyzer error: MESSx measurement is canceled</td>
<td>− equipment fault</td>
<td>− initialize analyzer</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>− check window System state for error removal</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>− search for equipment fault and remove error</td>
</tr>
<tr>
<td></td>
<td>Peltier temperature outside range</td>
<td>− Peltier cooling insufficient</td>
<td>− Notify service <strong>Note</strong>: After successful repair a replacement of the water traps is recommended.</td>
</tr>
<tr>
<td></td>
<td>Minimum sample volume &gt; cup volume</td>
<td>for sample supply with sampler:</td>
<td>check configuration in the method:</td>
</tr>
<tr>
<td></td>
<td></td>
<td>− sample volume selected too large</td>
<td>− sample volume/rinsing volume</td>
</tr>
<tr>
<td></td>
<td></td>
<td>− number of detections too high</td>
<td>− adjust number of detections (repeat measurements) to the cup volume</td>
</tr>
</tbody>
</table>
## 9.3 Status errors – indications in the window System state

### Note:
Status errors are shown in the window System state in red or yellow.

<table>
<thead>
<tr>
<th>Error indication</th>
<th>Remedy</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Flow indication MFC: 120 ml/min</strong>&lt;br&gt;Flow indication MFM: &lt; 110 ml/min</td>
<td></td>
</tr>
<tr>
<td><strong>Cause</strong></td>
<td><strong>Remedy</strong></td>
</tr>
<tr>
<td>− union nut at the combustion tube not tightened correctly (after catalyst replacement)</td>
<td>− check screw connections for completeness, deformation, tighten if necessary</td>
</tr>
<tr>
<td>− carrier gas supply to the furnace head not connected properly (after catalyst replacement)</td>
<td>− check carrier gas supply (FAST connector at the analyzer wall and screw connection at the furnace head)</td>
</tr>
<tr>
<td>− sealing rings at the combustion tube faulty (severely deformed) or not attached (after catalyst replacement)</td>
<td>− check all connection points (water traps) and replace if necessary</td>
</tr>
<tr>
<td>− TIC condensate container FAST connector leaking</td>
<td></td>
</tr>
<tr>
<td>− Loose connection at the water trap system (after installing water traps, installing halogen trap)</td>
<td></td>
</tr>
<tr>
<td>− connection between combustion tube and condensation coil or screw connections leaking</td>
<td>− check connection between combustion tube and condensation coil (tight fit of the fork clamp)</td>
</tr>
<tr>
<td>− combustion tube faulty (cracks, fractures at the edge)</td>
<td>− check glass components, replace if faulty</td>
</tr>
<tr>
<td>− TIC condensate faulty (fractures at the connections)</td>
<td></td>
</tr>
<tr>
<td>− Water traps clogged</td>
<td>− Replacing the water traps</td>
</tr>
<tr>
<td>− condensate pump hose leaking</td>
<td>− check condensate pump, replace hose if necessary</td>
</tr>
</tbody>
</table>

| **Flow indication MFC: 120 ml/min**<br>Flow indication MFM 1: < 110 ml/min or > 130 ml/min | |
| **Cause** | **Remedy** |
| − MFM (mass flow sensor) faulty | − check flow with external mass flow sensor to confirm error if possible |
| − halogen trap filling used up | − inform Service |
| | − check halogen trap |

| **Flow indication MFC: < 120 ml/min or fluctuating**<br>Flow indication MFM1 : < 110 ml/min | |
| **Cause** | **Remedy** |
| − no carrier gas | − turn on carrier gas at the pressure reducer |
| − hose line leaking | − 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 106

− MFC faulty

− inform Service

**Flow indication MFC: 120 ml/min**

**Flow indication MFM 1: > 130 ml/min**

<table>
<thead>
<tr>
<th>Cause</th>
<th>Remedy</th>
</tr>
</thead>
<tbody>
<tr>
<td>Peltier cooling insufficient (simultaneous error message temperature out of range below Peltier temperature)</td>
<td>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)</td>
</tr>
<tr>
<td>MFC faulty</td>
<td>inform Service</td>
</tr>
</tbody>
</table>

**Flow indication MFC: 0 ml/min**

**Flow indication MFM 1: 0 ml/min**

<table>
<thead>
<tr>
<th>Cause</th>
<th>Remedy</th>
</tr>
</thead>
<tbody>
<tr>
<td>a hose line is clogged up</td>
<td>replace clogged hose line</td>
</tr>
<tr>
<td>if necessary remove and rinse clogged hose line, then reinstall</td>
<td></td>
</tr>
<tr>
<td>no method loaded</td>
<td>load method</td>
</tr>
</tbody>
</table>

**Values of the NDIR detector below opt. bank indicated in yellow**

<table>
<thead>
<tr>
<th>Cause</th>
<th>Remedy</th>
</tr>
</thead>
<tbody>
<tr>
<td>ADU values of the NDIR detector differ from the target value</td>
<td>check halogen trap and replace if necessary</td>
</tr>
<tr>
<td>consult regarding the application with Analytik Jena AG about specific application rules for difficult sample matrix</td>
<td></td>
</tr>
</tbody>
</table>

*Note:* 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.**

*Note:* 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

---

**CAUTION**

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!
9.4 Equipment faults and analytical problems

Other problems not detected by the system monitoring can also occur. 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.

<table>
<thead>
<tr>
<th>Error</th>
<th>Remedy</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water traps clogged</td>
<td></td>
</tr>
<tr>
<td><strong>Cause</strong></td>
<td><strong>Remedy</strong></td>
</tr>
<tr>
<td>- Lifetime exceeded (renewal after 6 month recommended, matrix depend-ent)</td>
<td>Replacement of the water traps (see section &quot;Replacing the water traps&quot; p. 82)</td>
</tr>
<tr>
<td>- Measuring of samples with strong aerosol generation</td>
<td></td>
</tr>
</tbody>
</table>

| Scattering measured values | |
| **Cause** | **Remedy** |
| - Filling of combustion tube depleted | - Perform catalyst change |
| - metering faulty | - check metering |
| - inhomogeneous sample matrix | - Warm up cold samples before analysis |
| | - filter samples prior to analysis |
| - sensitive samples | - prevent introduction of CO₂ or organic vapors from the ambient air |
| | - cover sample cups on sampler with aluminum foil |
| | - Aerate the head room of the sample container for manual measurements |
| | - check the environmental conditions |
| | - remove the source of interference |
| drift NDIR basic | |
| - unsuitable integration criteria | - check the settings |
| - measurement is canceled too early | - increase the maximum integration time |

| Sample is not drawn up without air bubbles | |
| **Cause** | **Remedy** |
| - leaks in the sample intake path | - check connections and tighten any loose connections: cannula – hose |
| | - hose – syringe pump valve |
| - sample intake cannula clogged | - remove the cannula and clean in an ultrasonic bath |
| | - replace cannula |
| - metering syringe leaking | - remove and check metering syringe |
| - PTFE sealing lips of the plunger are damaged | - replace metering syringe |

| incomplete metering in reactors | |
| **Cause** | **Remedy** |
| - leaks in the metering path | - check connections and tighten any loose connections: syringe pump – change-over valve |
### Fault removal

| Carry-over | change-over valve – injection cannula  
|------------|------------------------------------------|
|            | change-over valve – TIC condensate  
|            | vessel                                   |

#### Cause

<table>
<thead>
<tr>
<th>Cause</th>
<th>Remedy</th>
</tr>
</thead>
<tbody>
<tr>
<td>insufficient syringe rinse</td>
<td>rinse the metering syringe with sample</td>
</tr>
<tr>
<td></td>
<td>before the next injection: under Method</td>
</tr>
<tr>
<td></td>
<td>/ Edit in the tab Method enter 3 for rinse</td>
</tr>
<tr>
<td></td>
<td>cycles for the first measurement,</td>
</tr>
<tr>
<td></td>
<td>for all other measurements rinsing is</td>
</tr>
<tr>
<td></td>
<td>generally not required, enter 0</td>
</tr>
</tbody>
</table>

#### Low results; all areas

<table>
<thead>
<tr>
<th>Cause</th>
<th>Remedy</th>
</tr>
</thead>
<tbody>
<tr>
<td>Catalyst depleted</td>
<td>Exchanging the catalyst</td>
</tr>
<tr>
<td>system is leaking</td>
<td>inspect system for leaks</td>
</tr>
<tr>
<td>faulty metering</td>
<td>check metering</td>
</tr>
</tbody>
</table>

#### Low results for analyses through combustion (TC, TOC, NPOC, TN_b)

TIC measurements are ok

<table>
<thead>
<tr>
<th>Cause</th>
<th>Remedy</th>
</tr>
</thead>
<tbody>
<tr>
<td>Catalyst depleted</td>
<td>During measurements in the differential mode (neutral samples, slightly alka-</td>
</tr>
<tr>
<td></td>
<td>line) it is recommended to regularly regenerate the catalyst:</td>
</tr>
<tr>
<td></td>
<td>Multiple injection (6 times) of acidified ultrapure water (with HCl to pH 2)</td>
</tr>
<tr>
<td></td>
<td>Replace catalyst if regeneration without or only with brief success</td>
</tr>
<tr>
<td></td>
<td>For measurements in NPOC mode (acidic samples) always replace the catalyst</td>
</tr>
<tr>
<td></td>
<td><strong>Note:</strong> After a catalyst replacement a calibration must be carried out.</td>
</tr>
</tbody>
</table>

#### Low results for TIC measurements

Analyses through combustion (TC, TOC, NPOC) are ok

<table>
<thead>
<tr>
<th>Cause</th>
<th>Remedy</th>
</tr>
</thead>
<tbody>
<tr>
<td>no phosphoric acid in the reagent bottle</td>
<td>refill phosphoric acid</td>
</tr>
<tr>
<td>for phosphoric acid</td>
<td></td>
</tr>
<tr>
<td>faulty metering of the sample</td>
<td>check metering</td>
</tr>
</tbody>
</table>

#### Low results for TN_b

<table>
<thead>
<tr>
<th>Cause</th>
<th>Remedy</th>
</tr>
</thead>
<tbody>
<tr>
<td>Catalyst depleted</td>
<td>Exchanging the catalyst</td>
</tr>
<tr>
<td>measurement outside the calibrated range</td>
<td>Pay attention to calibrated range</td>
</tr>
<tr>
<td></td>
<td>Calibrate dependent on matrix when possible</td>
</tr>
<tr>
<td></td>
<td>when analyzing unknown substances</td>
</tr>
<tr>
<td></td>
<td>use low concentration where possible</td>
</tr>
<tr>
<td></td>
<td>(dilute sample if possible)</td>
</tr>
</tbody>
</table>
## Unusual peak form (TC and TN$_b$-Measurement)

<table>
<thead>
<tr>
<th>Cause</th>
<th>Remedy</th>
</tr>
</thead>
<tbody>
<tr>
<td>Catalyst depleted</td>
<td>if low results simultaneously, replace catalyst</td>
</tr>
<tr>
<td>unfavorable integration criteria selected</td>
<td>check integration criteria</td>
</tr>
<tr>
<td>exceeding the measuring range for TN$_b$</td>
<td>Dilute samples</td>
</tr>
<tr>
<td>measurement with CLD (peak height &gt; 1000 ppm NO in the measuring gas)</td>
<td></td>
</tr>
</tbody>
</table>

## TN$_b$ measurements with CLD faulty (TC measurements are ok)

<table>
<thead>
<tr>
<th>Cause</th>
<th>Remedy</th>
</tr>
</thead>
<tbody>
<tr>
<td>gas connection between multi N/C pharma HT and CLD faulty</td>
<td>check gas connection between multi N/C pharma HT and CLD</td>
</tr>
<tr>
<td>ozone generator faulty</td>
<td>inform Service</td>
</tr>
</tbody>
</table>

## Condensate pump/phosphoric acid pump leaking

<table>
<thead>
<tr>
<th>Cause</th>
<th>Remedy</th>
</tr>
</thead>
<tbody>
<tr>
<td>leaking hose connections</td>
<td>replace the pump hose</td>
</tr>
<tr>
<td>defect pump hose</td>
<td></td>
</tr>
</tbody>
</table>

## Control lamps at the analyzer do not illuminate: 5 V, 24 V

<table>
<thead>
<tr>
<th>Cause</th>
<th>Remedy</th>
</tr>
</thead>
<tbody>
<tr>
<td>error in the power supply or in the electronics</td>
<td>check the electrical connections</td>
</tr>
<tr>
<td></td>
<td>check the power supply of the lab</td>
</tr>
<tr>
<td>equipment fuse faulty</td>
<td>inform Service</td>
</tr>
</tbody>
</table>

## Front LED display on the analyzer does not illuminate: Lockin

<table>
<thead>
<tr>
<th>Cause</th>
<th>Remedy</th>
</tr>
</thead>
<tbody>
<tr>
<td>internal program has not been started</td>
<td>switch analyzer on again (switch off/on from main switch)</td>
</tr>
</tbody>
</table>

## Control lamps at the analyzer do not illuminate: Heating

<table>
<thead>
<tr>
<th>Cause</th>
<th>Remedy</th>
</tr>
</thead>
<tbody>
<tr>
<td>incorrect temperature configuration in multiWin</td>
<td>check temperature configuration in multiWin under Configuration / Edit options on the tab Device components (list field furnace temperature)</td>
</tr>
<tr>
<td>faulty thermocouple (furnace) <em>Note</em>: A faulty thermocouple can be detected by an indication in the LED strip in the analyzer</td>
<td>inform Service</td>
</tr>
<tr>
<td>faulty electronics component</td>
<td>inform Service</td>
</tr>
<tr>
<td>combustion furnace not connected correctly</td>
<td>check correct contact of the combustion furnace</td>
</tr>
</tbody>
</table>
10 Transport and storage

10.1 Transport

10.1.1 Preparing the analyzer for transport

**CAUTION! RISK OF BURNING AT THE HOT FURNACE!**
There is a risk of burning on the combustion furnace! Only remove the combustion furnace when the device is cold or allow the device to cool down sufficiently!

**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!

**CAUTION! USE SUITABLE TRANSPORT PACKAGING!**
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!

**CAUTION!**
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 bottle and drip tray 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 TIC condensate vessel" p. 92).
8. Pack open hose ends in protective bags and secure them e.g. using adhesive tape.
9. Open the left side wall,
   - Unscrew the four fastening screws; the screws are undetachable and remain in the wall.
   - Disconnect the grounding conductor connection and put the side wall safely aside.
10. Carefully remove the condensation coil from the holder, drain the condensation coil and safely put it aside (see section "Removing and cleaning the condensation coil" p. 94).
11. Remove the combustion tube (see section "Removing the combustion furnace" p. 100).
12. Remove the combustion furnace (see section "Removing the combustion furnace" p. 100).
13. Package the hose end of the condensation coil in a protective bag and secure it with adhesive tape.
14. Remove the cannulas from the hoses and insert the cannulas into the cannula packaging.
15. Close the left side wall of the analyzer:
− Connect the grounding conductor connection to the side wall.
− First screw in the screws at the bottom and then the top side. Tighten the screws in turn.

16. Close the doors of the analyzer.
17. Attach the top cover and secure it with adhesive tape.
18. Carefully package the accessories, in particular protect glass components against breakage.

10.1.2 Transport notes

Observe the safety instructions in section "Safety instructions, transport and installation" p. 9. 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.

10.1.3 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. Place the autosampler on the side as shown in Fig. 48.
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. 48) with the Allen wrench.
10.1.4 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.
- 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. 38.

10.2 Storage

CAUTION
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
10.3 Recommissioning after transport or storage

10.3.1 Assembling the analyzer after transport or storage

When positioning the analyzer observe the notes in section "Site requirements" p. 38.

Assemble the components of the analyzer as follows:

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 at the top cover and the side walls, remove the top cover, put it safely aside, and open the doors.
4. Open the left side wall,
   - Unscrew the four fastening screws; the screws are undetachable and remain in the wall.
   - Disconnect the grounding conductor connection and put the side wall safely aside.
5. Remove all remaining adhesive tape and protective bags.
6. Install the combustion furnace (see section "Installing the combustion furnace" p. 102).
7. Install the halogen trap and the water traps (the water traps are connected to the halogen trap).
8. Install the TIC condensate vessel (see section "Cleaning the TIC condensate vessel" p. 92).
9. Install the condensation coil (see section "Inserting the condensation coil" S. 95).
10. Fill the combustion tube and install the combustion tube in the combustion furnace (see sections "Filling the combustion tube" p. 88 and "Inserting the combustion tube" p. 90).
11. Close the left side wall of the analyzer:
    - Connect the grounding conductor connection to the side wall.
    - First screw in the screws at the bottom and then the top side. Tighten the screws in turn.
12. Place the reagent bottle with the drip tray into the analyzer.
13. Connect the cannulas to the hoses no. 7 and AA and tighten the Fingertight connections finger-tight.
14. Close the doors of the analyzer.
15. Position any add-on devices at the intended location and connect them.
10.3.2 Connecting the analyzer

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

1. Main switch to switch the analyzer on and off "power switch"
2. Holder for mains fuse "FUSE"
3. Mains connection "main plug"
4. Gas connection "CLD"
5. Carrier gas connection "O₂ oxygen"
6. Connection of the neutral conductor at the sampler
7. Waste "waste"
8. RS 232 interface for the sampler "sampler"
9. RS 232 interface for CLD "CLD"
10. USB port for PC

![Diagram of mains connection and gas connections at the multi N/C pharma HT](image)

**Fig.49 Mains connection and gas connections at the multi N/C pharma HT**

**Connect the mains cable**

**CAUTION**
Always connect the electrical system components to the multi N/C pharma HT 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!

**CAUTION! DAMAGE FROM CONDENSATE!**
Settled condensation and temperature differences can damage individual components of the analyzer during recommissioning.
Allow the analyzer multi N/C pharma HT to acclimatize for at least one hour before commissioning after positioning it in the operating room.

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.49 p. 120).
2. Connect the mains plug of the low-heat connection cable to a grounded socket.
Connecting the gas supply

**IMPORTANT**

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:

1. Connect the connection hose supplied to the pressure reducer of the gas supply and the gas connection "O₂" on the equipment backplate (5 in Fig.49 p. 120).
2. 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:

1. Connect the waste hose to the "waste" connection at the analyzer backplate and lead the loose end into a suitable waste container or drain.
2. Open the right front door at the analyzer and place the reagent bottle filled with 10 % phosphoric acid and the drip tray into the analyzer.
3. Connect the hoses no. 4 and no. AC to the reagent bottle with phosphoric acid.
11 Disposal

11.1 Waste water

ENVIRONMENTAL PROTECTION

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

Waste water arises during the ongoing analysis operation of the multi N/C pharma HT. This water contains hydrochloric 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.

11.2 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.

11.3 Catalyst

ENVIRONMENTAL PROTECTION

Used catalyst should be disposed of in accordance with the local regulations (waste frame: used catalyst, CeO₂, Pt(Al₂O₃).

Analytik Jena AG accepts the special catalyst back for disposal. Please contact the customer service department (see inside front cover).

11.4 Analyzer

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