multi EA 4000

Elementary analyzer
C, S and Cl solids analysis

Operating manual

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1 Basic Information

1.1 User manual notes

The analyzer multi EA 4000 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 elementary 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. 11.

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

- ☐ Menu command, buttons, options etc. are indicated by SMALL CAPS.
- ☐ Menu commands of a command sequence are separated by slashes (/), e.g. METHOD / EDIT.
- ☐ Buttons are marked with square brackets, e.g. [SAVE].

1.2 Intended Use

The elementary analyzer multi EA 4000 is a device for the detection of the total carbon content and/or total sulfur content or for the detection of the total chlorine content in solid and paste-like samples. After appropriate sample preparation it can also be used for AOX detection.

The system is configured and expanded in accordance with the specific analytical requirements.

The analyzer multi EA 4000 is versatile in its application due to its robust modular design, ease of operation and expandability.

Waste management

The multi EA 4000 is particularly suited for TIC/TOC detection in waste as well as the total chlorine detection in surrogate fuels.

Environmental monitoring

The multi EA 4000 enables the simultaneous carbon and sulfur detection in sediment and soil analysis.

Power station

An important application area for the multi EA 4000 is both the carbon detection in carbon or ash residue and the detection of residual carbon in the ash of incineration plants. The chlorine detection in surrogate fuels is also of significance here.

Material testing

The multi EA 4000 enables a rapid sulfur and/or carbon detection in construction materials, such as cement or plaster, in ceramic materials or glass samples.

Research and teaching

Due to the many specific configuration options the multi EA 4000 is suitable for use in teaching and research.

The analyzer multi EA 4000 must only be used for the methods for the detection of total carbon and/or total sulfur content or the detection of the total chlorine content in solid, paste-like or liquid samples described in this user manual. 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 farm explosive mixtures. No concentrated acids may be analyzed with the analyzer.

The device must only be operated with the carrier gases oxygen 2.5 and argon 4.5 or better.

The operational safety of the analyzer EA 4000 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 on the title page.

1.3 Warranty and liability

The warranty duration and liability comply with the legal requirements and the provisions in the general terms and conditions of Analytik Jena 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 EA 4000 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

2 Technical data

General characteristics	
Designation/type	Analyzer multi EA 4000
Dimensions	Basic device (W x H x D) 550 x 500 x 550 mm per module: 300 x 500 x 550 mm Sampler: 520 x 500 x 700 mm
Mass	approx. 35 kg
Procedural data	
Digestion principle	oxidative combustion
Digestion temperature	dependent on application 1000 – 1500 °C (lower temperatures also possible)
Measuring method	TC, TIC, TOC, TS, EC, TCl
C and S detection	NDIR (coupled with VITA method)
Cl detection	Coulometry
Sample volume	Up to 3000 mg
Sample Feed	Weighted sample in porcelain and quartz glass boats Automatic sampler or manual feed
Gas supply	Oxygen min. 2.5 and argon 4.5 with system pressure 2 – 4 bar
Control/analysis (control and analysis software multiWin)	Real-time graphics, status indication during analysis, graphical display of the measured results, result print-out
Electrical variables	
Connection	230 V AC (+ 10%, -15%), 50/60 Hz
Protection	T16 A H
Typical average power consumption	3700 VA
PC interface	USB
Interference suppression (electromagnetic compatibility)	in accordance with the provisions of EN 55022 class A interference-protected and BMPT version 243/1991
Environmental conditions	
Temperature during storage	5 – 55 °C
Temperature during operation	10 – 35 °C
Humidity during operation	max. 90% at +30 °C
Humidity during storage	(10 – 30) % (use desiccant)
Air pressure	0.7 1.06 bar

Minimum equipment for the control and analysis unit

Operating system:	Windows XP Professional with Service Pack 2.0 or better
Processor:	Pentium IV or better
Working memory:	2 GB
Free hard disk space:	40 GB
Drive:	CD-ROM drive
Monitor resolution:	1024 x 768
Interfaces	USB 2.0

Technical data for the Cl module

Measuring range (* with measuring gas splitting)	1 – 100 µg / 10 µg – 20 mg* absolute total chlorine
Furnace temperature	up to 1000 °C
Heat-up period	up to 30 min.
Analysis time	up to 10 min. – dependent on the combustion time and concentration
Sample weight (dependent on consistency and matrix)	Up to 3000 mg

3 Safety instructions

For your own safety and to ensure error-free and safe operation of the analyzer multi EA 4000, 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 multiWin control and analysis software on the monitor.

3.1 Standards and directives

The analyzer was manufactured according to the currently valid technology regulations and the approved safety related regulations.

During the construction of the analyzer the relevant safety and health requirements of the applicable laws, standards and regulations were applied. The safety of the analyzer is confirmed by the CE mark and the declaration of conformity.

Information regarding safety corresponds to the currently valid regulations of the European Union. In other countries the applicable laws and country specific regulations have to be complied with.

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

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

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

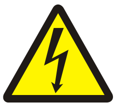
Indicates a potentially hazardous situation.
If it is not prevented death or most serious injuries (incapacitation) can result.

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.



WARNING! DANGER OF ELECTRIC SHOCK IF TOUCHED!

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.

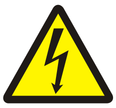
3.3 Safety markings on the analyzer

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:

Warning against hot surface



Warning against dangerous electrical voltage

Warning against hand injury from crushing

Warning against substances hazardous to health

Warning against corrosive substances

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 EA 4000 must only be operated by qualified specialist personnel instructed in the use of the analyzer. The instruction must also include conveying the content of this user manual and the user manuals of other system components or add-on equipment.

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

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

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

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

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

The 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 damaged to health the following must be observed when moving the analyzer in the laboratory (lifting and carrying):
  - For reasons of safety 2 persons are required to transport the analyzer and must position themselves on both sides of the equipment.
  - Because the analyzer does not feature any handles, firmly grip the device from the bottom and make sure prior to simultaneous lifting the device that the sensitive components at the front are protected by the closed doors.
  - The guide values and statutory limits for lifting and carrying loads without auxiliary equipment must be observed and adhered to.

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 decommissioned during operation.
- ☐ Free access to the power switch on the back of the enclosure has to be ensured during operation.
- ☐ The ventilation equipment on the multi EA 4000 and the extension modules must be in good working condition. Covered vents or ventilation slits etc. may cause the device to break down or may cause damage to it.
- ☐ The analyzer must not be switched on without the combustion tube being fitted.
- ☐ The furnace operates with temperatures of up to 1500 °C. The hot components (furnace, gas sluice, boat) must not be touched during or directly after the operation of the analyzer.
  In manual operation the hot boats must be deposited on the surface of the manual feed or on some other heat-resistant surface.
- ☐ During automatic cooling do not interrupt the power supply. In case of prolonged power failure during operation disconnect the mains plug and open the housing wall to prevent damage from overheating.
- ☐ 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 has 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 wit the applicable electrical engineering rules. Life-threatening electrical voltages may occur in the interior of the analyzer!

The following has to be observed:

- Extension modules or system components must always be connected to or disconnected from the analyzer in a deactivated condition.
- Before opening the analyzer it must be switched off from the equipment switch and the mains connector must be disconnected from the mains outlet!
- The analyzer must not be switched on without the combustion tube being fitted.
- 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 gases (argon and/or oxygen) are taken from compressed gas containers or local compressed gas systems. The required purity of the gas must be ensured (→ see chapter "Technical data" p.9)!

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 EC safety data sheets of the manufacturers for the auxiliary and operating materials.

The following has to be observed:

- ☐ The relevant regulations and the notes in the EC safety data sheets of the manufacturers have to be observed and complied with regards to storage, handing, use and disposal for all auxiliary and operation materials used during operation or maintenance of the analyzer.
- ☐ Auxiliary and operation materials may never be placed in containers or vessels for food. The approved containers for the relevant material are to be used and these have to be 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.
- ☐ Biological samples have to be handled according to local guidelines regarding the handling of infectious material.
- ☐ Caution when handing quartz glass and glass parts. Risk of broken glass and therefore risk of injury!
- ☐ Auxiliary and operating materials as well as their containers may not be disposed in domestic waste or enter the sewage system or the soil. The applicable regulations for disposal of these materials must be meticulously observed.
- ☐ Ensure good room ventilation in working rooms.

Handling concentrated acids

The following acids may be used during analysis:

- ☐ concentrated sulfuric acid as desiccant during Cl detection
- ☐ highly concentrated acetic acid and nitric acid for the preparation of the electrolytes during Cl detection
- ☐ ortho-phosphoric acid during TIC detection
- ☐ hydrochloric acid during TOC detection

Always observe the appropriate regulations and the notes in the EC safety data sheets of the manufacturers with regard to storage, use and disposal.

When handling the above-mentioned concentrated acids, observe in particular the following notes:

- ☐ The concentrated acid can cause burns and irritation!
- ☐ Always wear goggles and protective gloves when handling concentrated acids!
- ☐ Ensure adequate ventilation in the laboratory rooms.

- The above-mentioned concentrated acids must not enter the sewage system, surface water or ground water! The applicable regulations for disposal must be meticulously observed.
- Only store the concentrated acids in their original containers! Observe the notes on the labels.

IMPORTANT

In case of chemical burns from acid immediately carry out the following first aid measures:

- for skin burns:

  Immediately remove contaminated clothing and shoes, dab affected skin areas with polyethylene glycol 400 then rinse thoroughly with plenty of water.
- for eye burns:

  Rinse the eyes with open eye lid several minutes with running water (min. 10 minutes), then immediately consult a physician (even if there are no apparent symptoms).
- for burns of the respiratory organs:

  Bring the affected person into fresh air.
- if swallowed:

  Give plenty of water to drink (possibly several liters). Avoid vomiting (perforation risk). Do not attempt neutralization.

Always immediately consult a physician after carrying out the first aid measures!

Handling other substances for the preparation of electrolytes

For the preparation of the electrolyte solutions thymol, thymol blue, gelatin and methanol are used. Always observe the appropriate regulations and the notes in the EC safety data sheets of the manufacturer with regard to the storage, use and disposal of the substances.

Methanol

When handling methanol observe in particular the following notes:

- Methanol is toxic when inhaled, swallowed and in contact with the skin! There is a danger of irreversible damage!
- Always wear goggles and protective gloves when handling methanol!
- Provide eye wash bottles at the workplace.
- Ensure adequate ventilation in the laboratory rooms.
- Methanol is easily flammable! Keep methanol away from sources of ignition and take measures against electrostatic charges.
- Methanol must not enter the sewage system, surface water or ground water! The applicable regulations for disposal must be meticulously observed.
- Only store the methanol well sealed in its original container! Observe the notes on the labels.

IMPORTANT

In case of damage from methanol carry out the following first aid measures without delay (ensure your own protection first):

- after skin contact:

  Remove the contaminated clothing, rinse the affected skin areas with plenty of water.

- after eye contact:

  Rinse the eyes with the eye lids open with plenty of water, then immediately consult an optician.

- after inhalation:

  Bring the affected person into fresh air. In case of respiratory failure apply artificial ventilation immediately, supply oxygen where necessary. Consult a physician immediately.

- after swallowing:

  Give plenty of water to drink (possibly several liters). Induce vomiting. Give ethanol to drink (e. g. 1 glass of a 40% alcoholic drink). Consult a physician and point out methanol.

Always immediately consult a physician after carrying out the first aid measures!

Thymol

When handling thymol observe in particular the following notes:

- Thymol is hazardous to health when swallowed (chemical burns)!
- Provide eye wash bottles at the workplace.
- Ensure adequate ventilation in the laboratory rooms.
- Thymol is toxic to water-borne organisms and can cause long-term damage in waterways!
- Thymol must not enter the sewage system, surface water or ground water! The applicable regulations for disposal must be meticulously observed.
- Only store thymol sealed in its original container! Observe the notes on the labels.

IMPORTANT

In case of contact with thymol immediately carry out the following first aid measures:

- after skin contact:

  Immediately remove contaminated clothing, dab affected skin areas with polyethylene glycol 400 then rinse thoroughly with plenty of water.

- after eye contact:

  Rinse the eyes with open eye lid several minutes with running water (min. 10 minutes), then immediately consult a physician (even if there are no apparent symptoms).

- after inhalation:

  Bring the affected person into fresh air.

- after swallowing:

  Give plenty of water to drink (possibly several liters). Avoid vomiting (perforation risk). Do not attempt neutralization.

Always immediately consult a physician after carrying out the first aid measures!

Gelatin, Thymol blue

Gelatin and thymol blue are not hazardous substances in the meaning of the EC directives.

Carry out the following first aid measures in case of contact with these substances:

- after inhalation:

  Bring the affected person into fresh air.

- after skin contact:

  Rinse with plenty of water, take off contaminated clothing.

- after eye contact:

  Rinse with plenty of water with open lids.

- after swallowing:

  Give plenty of water to drink. In case of sickness consult a physician.

Formation of acetic acid vapor

During the chlorine analysis acetic acid vapor may form. This accumulates during the gas phase above the electrolyte surface (titration cell) or in the module housing.

Acetic acid vapor is not only corrosive but also hazardous to health.

Do not inhale these vapors! Always connect the adsorption tube with active carbon in the chlorine module without delay!

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 maladjust or damaged the analyzer. Therefore, the operator may generally only carry out the tasks listed in chapter 'Maintenance and care' p. 84.

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, service of the dust trap) 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 v 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 main switch (in the multi EA on the front panel, in the modules on the equipment backplate) and the mains plug must be disconnected from the mains outlet 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 System design

4.1 Comprehensive overview

The analyzer multi EA 4000 is of modular design. The basic device (combustion unit) is combined with one or several detector modules dependent on the task:

- ☐ Basic device
- ☐ Carbon and/or sulfur module (C/S module)
- ☐ Chlorine module (Cl module)
- ☐ TIC solids module (automatic or manual)

The change from C/S to Cl detection is possible without having to change the pneumatic and electrical connections. The ceramic tube is replaced by a combustion tube from quartz glass for this purpose.

The sample transfer to the combustion furnace is

- ☐ automatic using the sampler FPG 48 or
- ☐ manual using a manual feed.

The device system can be combined with scales with an interface to the control computer transferring the sample volume for calculating the analysis results automatically.

The control electronics is located in a detector module in accordance with the basic configuration.

- ☐ in the C/S module, if a C/S module is connected
- ☐ in the Cl module, if the analyzer has not been combined with a C/S module.

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.

4.2 Basic device

The basic device of the multi EA 4000 consists of the following main components:

- ☐ Combustion system
- ☐ Pneumatic and hose system
- ☐ Components for measuring gas drying and cleaning
- ☐ Indicator and control elements, connections

4.2.1 Combustion system

The combustion furnace is a resistor-heated horizontal furnace for digestion temperatures up to 1500 °C. A combustion tube is used as reactor. The carrier and combustion gas is supplied via an open gas airlock to the inside of the combustion tube. Since the gas flow of the carrier and combustion gas is greater than the intake flow of the measuring gas, the resulting overpressure at the inlet of the combustion tube prevents the measuring gas from escaping or ambient air from entering the combustion tube.

The combustion tube for the C/S detection consists of a special high temperature ceramic (HTC). This is particularly robust, corrosion-resistant and low in wear. Thanks to the HTC technology samples can be digested without catalysts at temperatures of up to 1500 °C. For particularly difficult matrices the combustion temperature can be increased to 1800 °C with special additives (utilization of the exothermal effect).

A quartz tube is used as combustion tube for the Cl detection. This features special chemical inertia against HCl and prevents too low results and memory effects during analysis.

The combustion system is equipped with a temperature controller monitoring the heating of the furnace, keeping the temperature at the target value and preventing overheating.

Horizontal furnace

Open gas airlock

Fig. 1 **Horizontal furnace of the basic device multi EA 4000**

4.2.2 Pneumatic and hose system

Hose system

The connection between the individual components and to the analysis modules is made via a hose system. The design of the hose system in the respective module configurations can be found in the hose diagrams in section "Recommissioning after transport or storage" p. 122 ff.

Controlling the gas flows

The gas flow for carrier gas and combustion gas is controlled via manually adjustable flow meters at the front of the basic device.

An integrated intake pump ensures the stable flow of the measuring gas through the detector. The necessary intake flow is set with the "pump" valve at the flow meter.

The pump and the gas flows are added automatically once the multiWin program has been started and the actual temperature of the combustion furnace differs less than 50 K from the target temperature. If the temperature differs more than 50 K from the target temperature, the pump and gas flows are automatically switched off.

1	flow meter for argon or oxygen "Ar/O_2"
2	flow meter for oxygen "O_2"
3	flow meter for intake flow "pump"

Fig. 2 **Flow meters at the basic device multi EA 4000**

4.2.3 Components for measuring gas drying and cleaning

Dust trap for C/S detection

The dust trap is attached directly to the ceramic combustion tube. Combustion dust settles inside it. Larger particles in the gas flow hit against a plate positioned vertically to the gas flow and fall down.

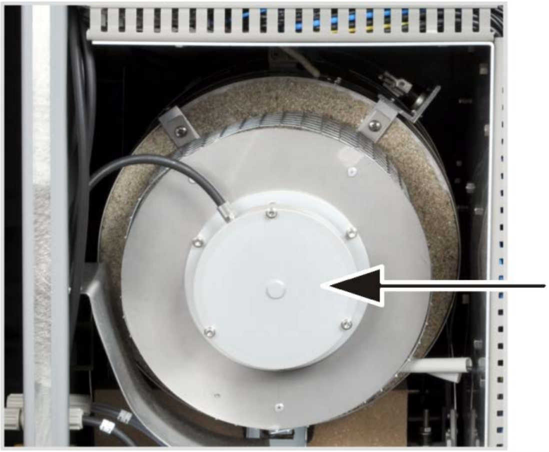
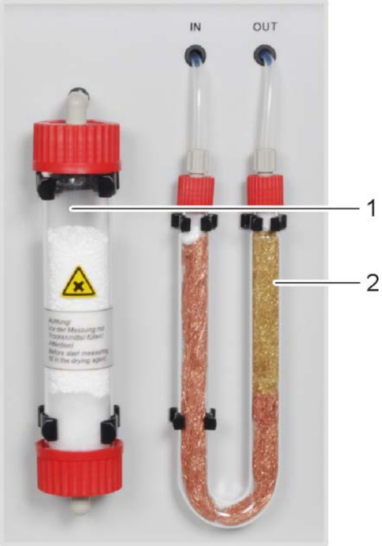


Fig. 3 **Dust trap at the combustion furnace**

Drying tube and halide trap for C/S detection

For drying the measuring gas during C/S the drying tube, filled with magnesium perchlorate as desiccant, has been installed in the gas path.

The basic device contains a halide trap (U tube) 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 halide trap has to be renewed at the latest once half of the copper wool or the brass wool is discolored.



1 drying tube with magnesium perchlorate

2 halide trap

Fig. 4 **Drying tube and halide trap**

4.2.4 Indicator and control elements, connections

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

Fig. 5 **Status indicators at the basic device multi EA 4000**

Behind the front doors there are the mains switch and the flow meters for the manual control of the gas flows.

1 flow meter for controlling the argon or oxygen flow "Ar/O_2"

2 flow meter for controlling the oxygen flow "O_2"

3 flow meter for controlling the intake flow "pump"

4 mains switch

Fig. 6 **Control elements behind the front doors of the multi EA 4000**

1 channel for measuring gas and by-pass hoses during Cl measurements

2 connection "Ar" for the connection to the combustion tube/gas airlock

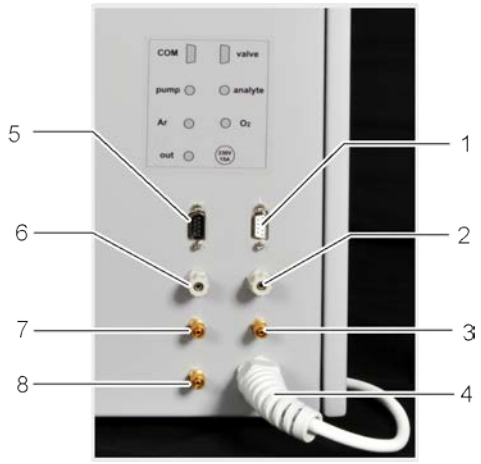
3 connection "O_2" for the connection to the combustion tube/gas airlock/TIC reactor

4 clip for securing the oxygen hose

5 opening of the combustion furnace with airlock coupling for combustion tubes

Fig. 7 **Connections on the right-hand side of the multi EA 4000**

On the rear of the multi EA 4000 are the gas inlets for the oxygen and argon supply, the connections for the attachment to the C/S or Cl module and the mains connection cable.



1	"valve" connection to the C/S module
2	"analyte" connection to the C/S module
3	gas connection "O_2" for oxygen
4	mains cable
5	"COM" connection to the C/S module or Cl module
6	"pump" connection to the C/S module or Cl module
7	gas connection "Ar" for argon during Cl measurements and pyrolysis
8	"out" connection – measuring output

Fig. 8 **Connections on the rear of the multi EA 4000**

4.3 C/S module

C/S module versions

The C/S detector module is available in three versions:

☐ the C/S module with

CO_2 and SO_2 detectors for the simultaneous detection of carbon and sulfur

☐ the C/S module with

CO_2 detector for the detection of carbon

☐ the C/S module with

SO_2 detector for the detection of sulfur

The control electronics is always integrated into the C/S module.

Detectors

The NDIR detectors (non-dispersive infrared absorption detector) are behind the rear right-hand side panel of the analyzer.

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

The detectors used in the C/S module are selective for CO_2 and SO_2.

Measurements using the VITA method

The CO_2/SO_2 molecules are detected by the measuring technology as long as they remain in the cell of the NDIR detector. Fluctuations of the measuring gas flow during the CO_2/SO_2 measurement lead to CO_2/SO_2 molecules sometimes being detected 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.

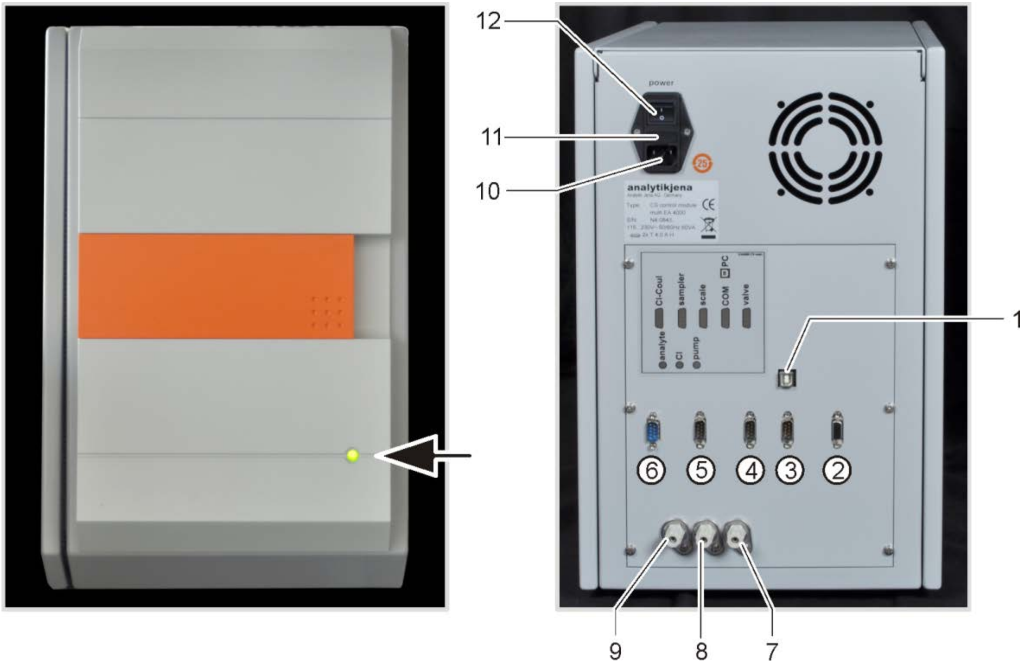
To detect the measuring gas flow a flow sensor is arranged very close to the NDIR detector flow.

Gas flow control

A flow meter monitors the necessary intake flow; the values are captured in the software. Deviations from the permissible target value are indicated, and the intake flow can be corrected at the "pump" valve of the flow meter of the basic device multi EA 4000 (3 in Fig. 6). During a measurement the values are used for the integrated correction using the VITA method.

Connections

The green LED located at the front of the C/S module illuminates once the module has been switched on. On the rear are the mains switch with mains input and fuse holder, the connections for attachment to the basic device and the additional add-on modules and the inlets and outlets for the measuring flows.



1	USB connection "PC" for connecting the computer
2	"valve" connection – connection to the multi EA 4000
3	"COM" connection – connection to the multi EA 4000
4	"scale" – connection of the scales
5	"sampler" connection – connection of the sampler FPG 48
6	Cl-Coul – connection to the Cl module
7	gas outlet "pump" – connection to the multi EA 4000
8	gas connection "Cl" – connection to the Cl module
9	gas inlet "analyte" – Measuring gas inlet
10	mains plug connection
11	fuse holder
12	mains switch

Fig. 9 **Indictors and connections at the C/S module**

4.4 Cl module

The Cl module consists of the following main components:

- Wide range coulometer for amperometry
- Stirring/cooling block for the measuring cells
- Measuring cells with electrodes
- Sulfuric acid container, possibly with splitting outlet and safety container
- Splitting valve
- Buffer container as pressure compensation to compensate the switching pulses of the splitting valve
- Adsorption tube
- Halide trap

The control electronics for the multi EA 4000 Cl is also located in the Cl module.

1	adsorption tube
2	halide trap
3	measuring cell with electrodes
4	stirring/cooling block

Fig. 10 **Cl module**

4.4.1 Coulometer assembly and measuring cell

The measuring cell is available in 2 versions:

	"sensitive" measuring cell 40 ml	**"high concentration" measuring cell 150 ml**
Electrolyte volume	approx. 20 ml	approx. 120 ml
Measuring range – absolute Cl (without measuring gas splitting)	1 – 100 µg	10 – 1000 µg
Measuring range (with measuring gas splitting)	1 – 2 mg abs. Total chlorine	10 – 20 mg abs. Total chlorine
Stirring principle	Magnetic stirring	Magnetic stirring

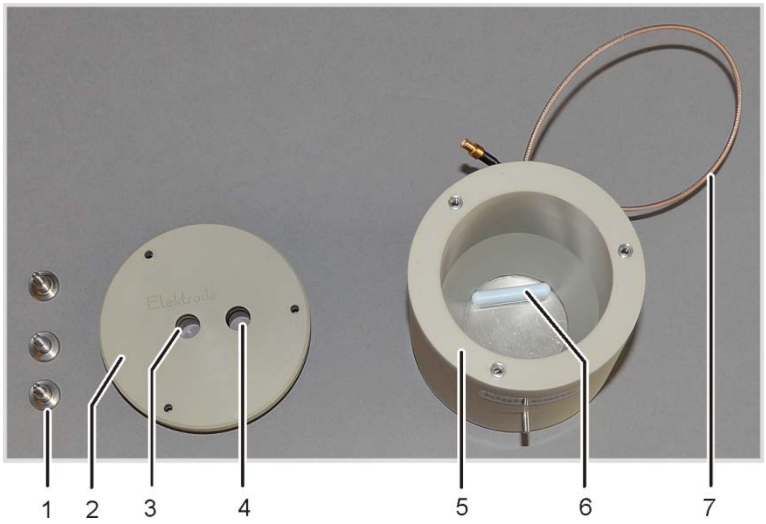
Indication principle	Alternating current bi-amperometry	Alternating current bi-amperometry
Generation principle	Anodic silver dissolution	Anodic silver dissolution
Generator current	Dynamic up to 1 mA	Dynamic up to 10 mA
Temperature control to recommended operating temperature	18 – 20 °C	18 – 20°C

Layout of the measuring cell

The measuring cell consists of the electrode space, containing the electrolyte solution, and the stirring block located in the module. At the floor of the electrode space is the generator anode in the form of a stable silver sheet. The magnetic stirrer runs above the anode.

The cell is sealed airtight with a cover and three knurled-head screws. The two cover lead-throughs (drill holes) support the combination electrode and a suction olive. The marked opening has been provided for the amperometric combination electrode. The unmarked opening is used for the direct injection into the measuring cell or for connecting the suction olive.

Switching on the chlorine module starts the movement of the magnetic stirring rod in the measuring cell cup. The preset cell temperature is 20 °C and can be modified as a method parameter in the multiWin program.



1	knurled-head screws
2	cover
3	opening for electrode (labeled)
4	opening for olive to connect a suction hose or direct injection
5	electrode space with silver anode
6	magnetic stirring rod
7	electrical connection of the measuring cell

Fig. 11 **Layout of the measuring cell**

Combination electrode

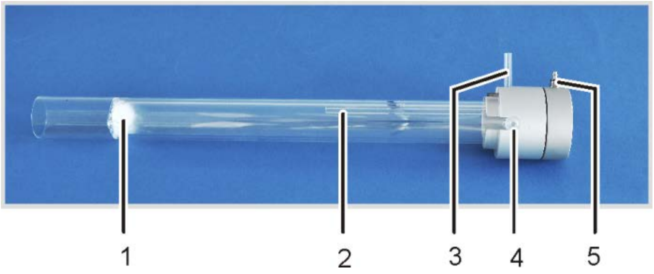
The combination electrode combines the indicator electrodes (Ag), generator cathode (Pt) and gas introduction. The generator anode is a round silver blank in the two measuring cell bodies (see above).

1	connector
2	measuring gas hose connection
3	gas introduction into the measuring cell
4	indicator electrodes (Ag)
5	generator cathode (Pt)

Fig. 12 **Combination electrode for measuring cell**

4.4.2 Combustion tube and measuring gas drying

A quartz tube is used as combustion tube during Cl detection. The oxygen for the combustion is supplied via a bypass in the quartz tube. The measuring gas is inducted at the side connection of the quartz tube and passed via the drying stage to the Cl module. The gas airlock, which is supplied with argon, prevents the measuring gas from escaping from the inlet of the combustion tube The end of the combustion tube is sealed with quartz wool to prevent dust from entering the hose system of the multi EA 4000.

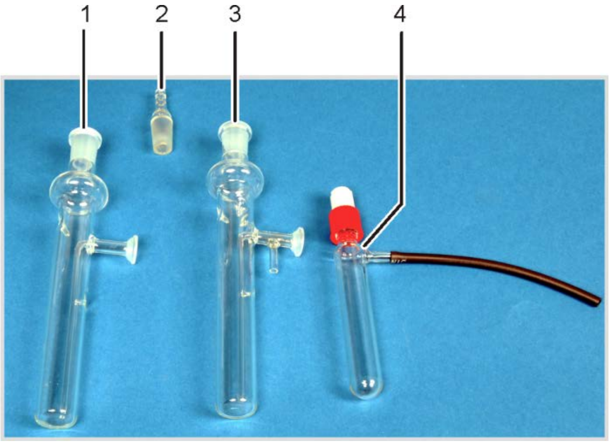


1	quartz wool plug	4	measuring gas outlet with boat connection
2	bypass for oxygen supply	5	argon supply at the gas airlock
3	connection socket for oxygen		

Fig. 13 **Quartz combustion tube with gas airlock**

Hydrogen chloride (HCl) has a high affinity for water and is therefore dissolved in its condensates. During analysis this behavior causes too low results or memory effects. To prevent condensation, the measuring gas must be dried immediately after it leaves the combustion tube. Concentrated sulfuric acid is used as a desiccant. Two special sulfuric acid containers are used dependent on whether the method includes measuring gas splitting or not.

When using the sulfuric acid container with split outlet, an additional safety container is installed in the gas path. This collects sulfuric acid that might be drawn out of the sulfuric acid container.



1	sulfuric acid container without split outlet
2	glass olive
3	sulfuric acid container with split outlet
4	safety container

Fig. 14 **Sulfuric acid containers for measuring gas drying**

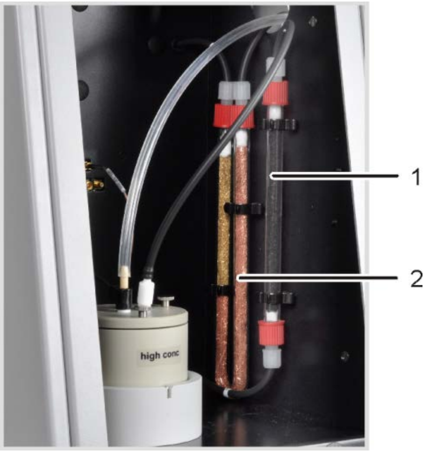
4.4.3 Measuring gas cleaning

Halide trap

In the operating mode with splitting the entire measuring gas from the combustion chamber is divided in a defined ratio. Only a part of the measuring gas reaches the measuring cell for analysis, allowing also for high chlorine concentrations to be detected. The discarded part of the measuring gas is passed via the halide trap in the Cl module to remove the HCl from the gas flow and protect the splitting valve and the intake pump against corrosion.

Adsorption tube

The gas extracted from the measuring cell is passed via the adsorption tube. Here the acetic acid vapors escaping from the electrolyte is removed. Active carbon is used as adsorption agent.



1	adsorption tube
2	halide trap

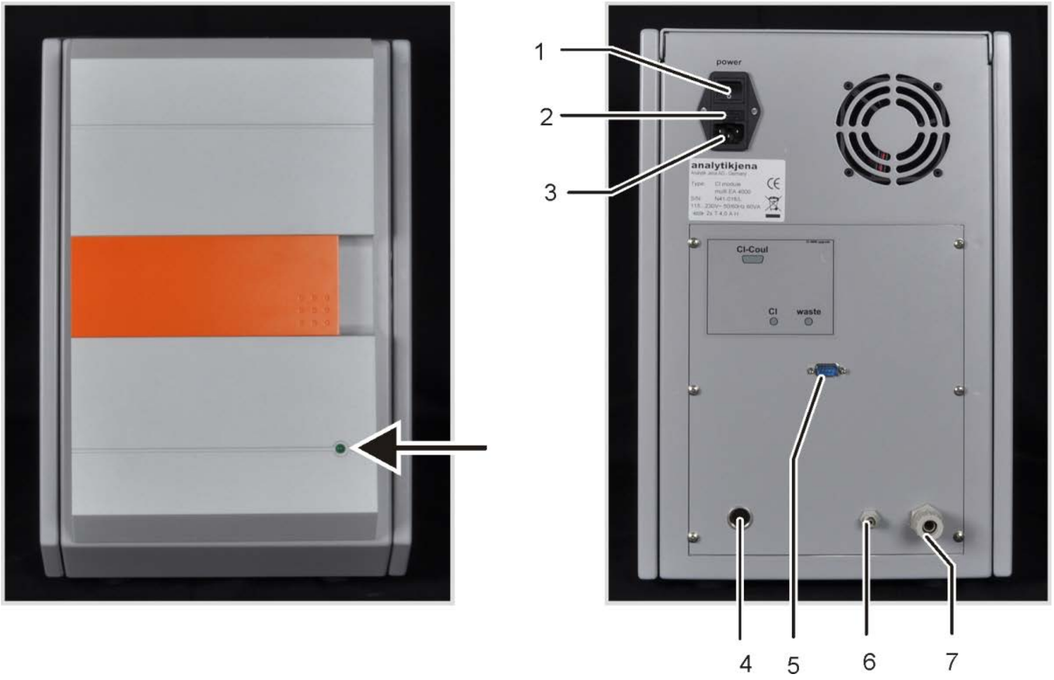
Fig. 15 **Halide trap and adsorption tube in the Cl module**

4.4.4 Connections

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

Dependent on the equipment of the multi EA 4000 with or without C/S module, the connection for attachment to the C/S module or the basic device and the sampler are found on the rear. The rear also features the outlet for the bypass in splitting mode.

The hoses for the supply of measuring gas and the bypass are passed through the duct in the basic device through the opening in the right-hand device panel to the chlorine module.



1 mains plug connection

2 fuse holder

3 mains switch

4 connection not allocated for the multi EA 4000

5 connection "Cl-Coul" – connection to the C/S module

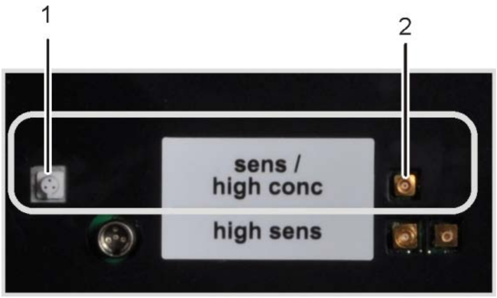
6 gas connection "Cl" – connection to the C/S module

7 gas outlet "waste"

Fig. 16 **Connections of the Cl module in combination with a C/S module**

Note:
If no C/S module has been installed, the Cl module has additional interfaces on the rear for the communication with the EA 4000 and the sampler. The interface allocation has been described in section "Installing the multi EA 4000 Cl" p. 125.

The electrical connections for the measuring cell and the combination electrode are located on the inside of the device backplate.



1 connection for combination electrode

2 connection of the measuring cell with silver anode

Fig. 17 **Connections for the coulometric cell at the inside panel of the Cl module**

4.5 Automatic TIC solids module

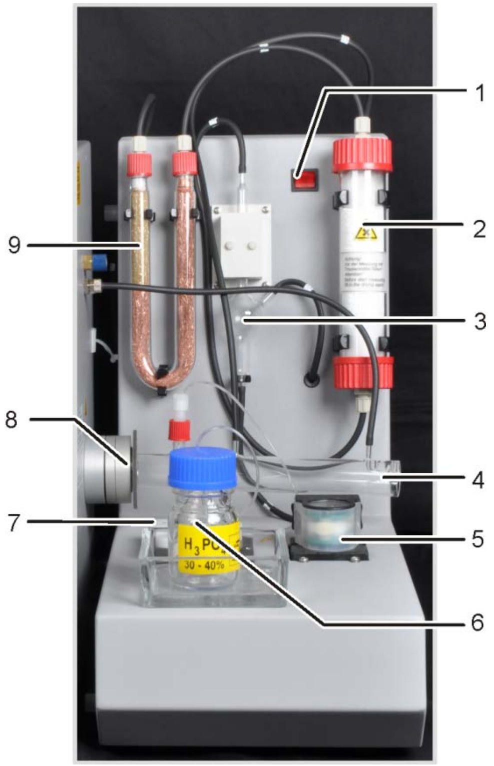
The automatic TIC solids module expands the multi EA 4000 for the detection of TIC (TC, TOC) in solids. Carbon dioxide generated by acidification and purging is passed to the NDR detector of the multi EA 4000. The following analysis modes are possible:

- TIC detection
- TOC detection in accordance with the differential method
- TOC detection in accordance with the direct method

The automatic TIC solids module is suited exclusively for automatic operation in combination with the multi EA 4000 C or multi EA 4000 C/S and the sampler FPG 48.

The automatic TIC solids module consists of the following components:

- TIC reactor
- phosphoric acid pump
- components for measuring gas cleaning and drying
- connections



1	mains switch
2	drying tube
3	condensate container
4	TIC reactor
5	condensate pump
6	bottle for phosphoric acid
7	acid pump for phosphoric acid or hydrochloric acid
8	connection rings of the TIC reactor to the multi EA 400
9	halide trap

Fig. 18 **Automatic TIC solids module**

4.5.1 Technical data

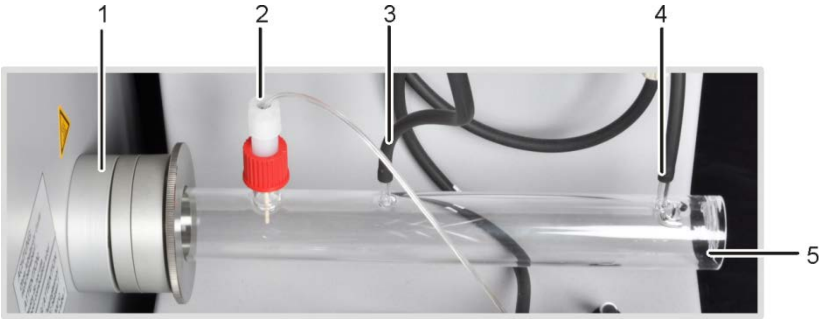
General characteristics	
Designation/type	Automatic TIC solids module
Dimensions	300 x 500 x 550 mm Sampler: 520 x 500 x 700 mm
Mass	approx. 5 kg

Procedural data	
Measuring method	TIC
Detection	NDIR (coupled with VITA method)
Sample volume	up to 3000 mg (TC) or 50 mg (TIC)
Electrical variables	
Connection	110 – 230V AC, 50/60 Hz
Protection	T 2 A H
Typical average power consumption	20 VA
Interface to the C/S module	RS 232

4.5.2 Layout

TIC reactor

The quartz tube is attached to the ceramic combustion tube with a coupling. A knurled ring with screw thread clamps the TIC reactor and seals the connection by pressure on the internal sealing rings. The reactor includes the connections for the acid supply, the measuring gas outlet and the oxygen supply. By adding the oxygen at the reactor inlet a gas airlock is implemented that prevents the measuring gas from escaping through the open inlet. The ceramic boats filled with samples are transferred to the reactor with the specially adapted sampler FPG 48.



1	holder at the multi EA 4000	4	inlet for oxygen supply
2	acid supply via ceramic cannula	5	insert for ceramic boats
3	measuring gas outlet		

Fig. 19 **TIC reactor**

Pneumatics/hose system

The connection between the individual components is made with labeled hoses. The hose diagram can be found in section "Installing the automatic TIC solids module" p. 132.

Hose pumps are used for the supply of the phosphoric acid and removal of the condensate. The phosphoric acid pump transports 40% phosphoric acid to the TIC reactor. Whilst the sample boat is moved continuously through the sampler FPG 48 towards the multi EA 4000, the sample is evenly diluted with phosphoric acid. Via the condensate pump the condensate is automatically pumped off after each measurement.

1 acid pump
2 condensate pump

Fig. 20 **Hose pumps at the automatic TIC solids module**

Components for measuring gas drying and cleaning

From the reactor the measuring gas is fed to the condensation coil attached to the rear of the automatic TIC solids module. The measuring gas is cooled down quickly in the condensation coil and the water vapor condenses. The measuring gas/water mixture is passed via a hose to the gas/liquid separator consisting of a condensate container and a Peltier cooling block.

The measuring gas is passed to the condensate container through the side entrance. The measuring gas is dried by freezing in the cooling block. The dry measuring gas is routed via the top connection out of the condensate container. After each measurement the condensate pump pumps the condensate draining through the bottom outlet automatically to the waste container.

The measuring gas is finally passed through the drying tube filled with magnesium perchlorate. This concludes the drying.

The gas path contains a halide trap (U tube) after the measuring gas drying process for the greatest possible removal of interfering components in the measuring gas and to protect the detectors and the flow meter in the C/S module. 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.

1	halide trap	4	drying tube
2	condensate container	5	condensation coil
3	Peltier cooling block		

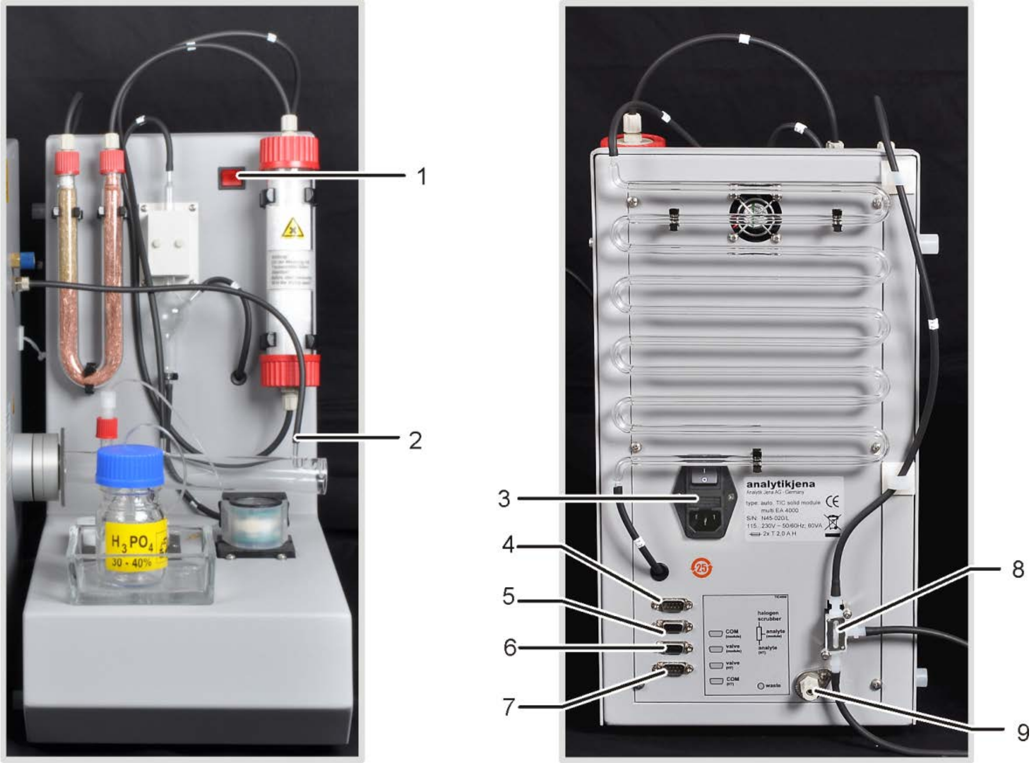
Fig. 21 **Measuring gas drying and cleaning at the automatic TIC solids module**

A dust filter is located between the change-over valve on the rear of the automatic TIC solids module and the measuring gas inlet of the C/S module.

Fig. 22 **Dust trap before the measuring gas inlet of the C/S module**

Connections

The connections are located on the rear of the automatic TIC solids module.



1	mains switch
2	gas connection for oxygen at the TIC reactor
3	mains supply module with mains switch and fuse holder
4	connection "COM module" for connection to the C/S module
5	connection "valve module" for connection to the C/S module
6	connection "valve HT" for connection to the multi EA 4000
5	connection "COM HT" for connection to the multi EA 4000
8	change-over valve
9	connection for the waste hose to drain the condensate

Fig. 23 **Connections at the automatic TIC solids module**

IMPORTANT

The automatic TIC solids module is equipped with two mains switches. Because the switch at the mains supply module below the condensate coil is poorly accessible, there is a second one at the front panel. Both mains switches must be switched on during operation. To switch the device off it is sufficient to actuate the switch on the front panel.

4.6 Manual TIC solids module

The manual TIC solids module enables the detection of TIC in solids in combination with the multi EA 4000 C or multi EA 4000 C/S. The acidification and purging of the TIC takes place at raised temperatures. The measuring gas is moved together with the carrier gas to the detector of the C/S module.

The manual TIC solids module is only designed for the manual sample supply. Automation using a sampler is not possible.

1	manual TIC solids module
2	C/S detector
3	basic device multi EA 4000

Fig. 24 **Manual TIC solids module**

4.6.1 Technical data

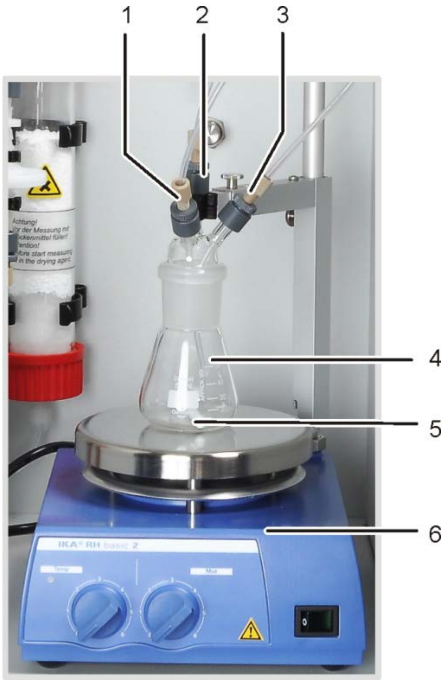
General characteristics	
Designation/type	Manual TIC solids module
Dimensions	300 x 500 x 550 mm
Mass	approx. 10 kg
Procedural data	
Measuring method	TIC
Detection	NDIR (coupled with VITA method)
Sample volume	Up to 3000 mg
Gas supply	Oxygen min. 2.5 2 – 4 bar
Electrical variables	
Connection	230 V AC (+ 10%, -15%), 50/60 Hz
Protection	T 16 A H
Typical average power consumption	3700 VA

4.6.2 Layout of the manual TIC solids module

TIC reaction chamber

The reactor is a 50 ml Erlenmeyer flask onto which a TIC head is placed with three connections for the supply of the phosphoric acid, the oxygen as carrier gas and the measuring gas.

A magnetic stirrer with heated working plate ensures a good mixing of the sample with the supplied acid and heats it slightly to accelerate the reaction.

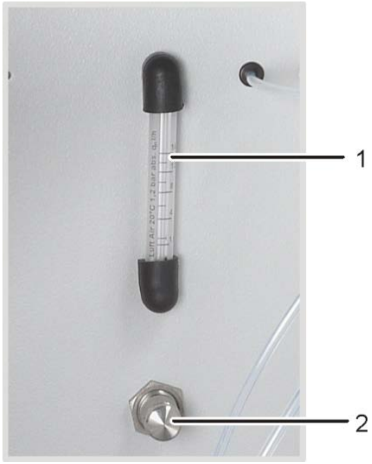


1	phosphoric acid supply (hose no. 6)
2	measuring gas outlet
3	carrier gas supply (oxygen)
4	Erlenmeyer flask
5	magnetic stirring rod
6	magnetic stirrer with heated working plate

Fig. 25 TIC reactor of the manual TIC solids module

Flow adjustment of the carrier gas flow

The carrier gas flow is adjusted via the needle valve "O2" with glass gauge on the rear of the manual TIC solids module.

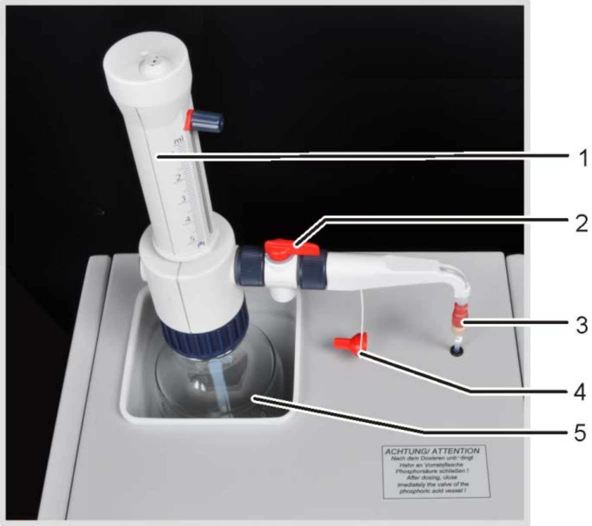


1	glass gauge
2	needle valve

Fig. 26 Flow meter for oxygen flow

Acid supply

The acid is metered manually using the metering pump. The hose for the acid supply to the reactor is connected through a valve to the metering pump. The valve in the cock of the metering pump must be closed after supplying acid to the reactor. This prevents the acid from accidentally being sucked into the analyzer.



1	metering pump with gradation
2	recirculation valve
3	valve for connecting the metering hose
4	sealing cap
5	phosphoric acid bottle

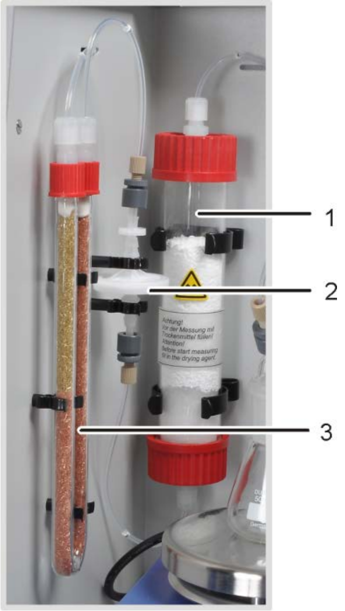
Fig. 27 Metering pump for acid at the manual TIC solids module

Measuring gas drying and cleaning

The drying tube, filled with magnesium perchlorate as desiccant, is installed in the gas path directly after the reactor.

The drying cartridge is followed by two water traps. The larger water trap (TC Pre-filter) retains aerosol during operation, the smaller water trap (disposable retention filter) retains rising water.

The C/S module contains a halide trap for the greatest possible removal of interfering components in the measuring gas and to protect the detector. The U-tube of the halide trap is filled with special copper wool and brass wool. The filling of the halide trap has to be renewed at the latest once half of the copper wool or the brass wool is discolored.



1	drying tube
2	water traps
3	halide trap

Fig. 28 **Components for measuring gas drying and cleaning at the manual TIC solids module**

Connections

1	mains supply module with mains switch and fuse holder
2	measuring gas outlet "analyte"
3	carrier gas connection "O_2"

Fig. 29 **Connections at the manual TIC solids module**

4.7 Other optional accessories

4.7.1 Sampler

Two sample supply versions are available for the multi EA 4000:

- ☐ the solids autosampler FPG 48
- ☐ the manual feed

Autosampler FPG 48

The autosampler FPG 48 has space for 48 sample boats. The boats are received automatically by the trays and transferred to the combustion furnace. The feed for the transfer is controlled by computer. Dependent on the equipment, the boats are redeposited on the sampler after measurement or thrown into a waste container after cooling.

For analyses in the quartz combustion tube (Cl measurements), the FPG 48 can be combined with a flame sensor.

Manual feed

The manual feed is a robust arrangement by which the ceramic hook with the sample boats can be kept straight and moved into the combustion furnace. During manual sample feed the transfer into the combustion tube must be slow and controlled for very reactive samples.

4.7.2 Flame sensor

For the Cl detection a flame sensor can optionally be combined with the sampler FPG 48. The flame sensor visually detects the combustion in the quartz tube for highly reactive samples and the boat feed is controlled in accordance with the brightness of the flame. The combustion characteristics optimized in this way lead to precise measuring results.

4.7.3 Scales

For the automatic volume transfer for calculating the analysis results, a scale can be connected. The samples must be weighed in accordance with the instructions in the program. The determined masses are automatically transferred to the sample table.

5 Measuring method

The analyzer multi EA 4000 is used to determine the following parameters as sum parameters.

- TS – Total Sulfur
- TC – Total Carbon
- TCl – Total Chlorine
- TOC – Total Organic Carbon
- TOC – Total Inorganic Carbon
- EC – Elemental Carbon
- TC-EC Carbon detection after pyrolysis
- AOC – Degradable Organic Carbon

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

5.1 TC/TS analysis

The multi EA 4000 with C/S module is a special system for the simultaneous and individual detection of total carbon and total sulfur in solid and paste-like samples through high temperature oxidation in the oxygen flow.

A sample aliquot is eight precisely into a combustion boat and transferred completely to the hot zone of the furnace. There it is dissolved and the sample oxidized in the oxygen flow at high temperature.

$$R + O_2 \rightarrow \mathbf{CO_2} + H_2O \tag{1}$$

$$R\text{-}S + O_2 \rightarrow \mathbf{SO_2} + CO_2 + H_2O \tag{2}$$

R carbonic substance

The resulting measuring gas is dried and halides it contains are removed. The CO_2/SO_2 content in the carrier gas is analyzed using NDR detectors which are selective for CO_2 and SO_2.

5.2 TIC analysis

In the TIC analysis the total inorganic carbon is detected in carbonates and hydrogen carbonates of solid and powdered samples. Cyanides, cyanates, isocyanates and carbon particles are not detected.

For the determination of the inorganic carbon (TIC) acid is added directly to an aliquot of the sample. This dissolves the carbonates/hydrogen carbonates it contains. The CO_2 is purged and detected. No thermal oxidation takes place.

$$3\,(CO_3)^{2-} + 2\,H_3PO_4 \rightarrow 3\,H_2O + 3\,CO_2 + 2(PO_4)^{3-} \qquad (3)$$

The sample digestion takes place in a TIC solids module. The measuring gas is detected in the C/S module.

5.3 TOC analysis

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

In the multi EA 4000 the TOC content can be determined in two ways:

☐ Differential method

☐ Direct method

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

IMPORTANT

For the TOC analysis using the automatic solids module the differential method should be preferred. The combustion tube and detectors are then not loaded with acid.

5.3.1 Differential method for the TOC analysis

The differential method can take place using the automatic TIC solids module. The sample is weight onto two boats. The TIC and TC are determined in consecutive measurements. The calculated difference is given as TOC. The differential method detects volatile as well as non-volatile organic carbon compounds.

$$TOC = TC - TIC \qquad (4)$$

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

5.3.2 Direct method for the TOC analysis

With the automatic TIC solids module the TIC and TOC can also be detected directly. After purging the TIC in the TIC reactor with **hydrochloric acid** and detecting the TIC content, the sample boat is transferred to the combustion tube. As in the TC/TS analysis, the sample is dissolved and oxidized in the oxygen flow at high temperature.

CAUTION

For the detection of TOC using the automatic TIC solids module hydrochloric acid has to be added to the sample. Therefore, the differential method is preferred for the TOC analysis.

Without using the automatic TIC solids module hydrochloric acid is added to the solid sample on the combustion boat to remove the inorganic bound oxygen. The CO_2 resulting from the reaction from the carbonates evaporates. The wet sample with added hydrochloric acid is dried in the drying cabinet at < 40 °C for at least 3 hours and the excess hydrochloric acid evaporates. The sample prepared in this way can then be examined in the analyzer for inorganic bound carbon as in the TC/TS analysis.

IMPORTANT

Because the acid only releases CO_2 from carbonates and hydrogen carbonates, elementary carbon or carbon from carbides, cyanides, cyanates, isocyanates and thiocyanates is also detected as TOC.

5.4 Carbon detection after pyrolysis: TC-EC (optional)

The detection of the carbon content of a sample is carried out in four steps in the optional pyrolysis mode.

The original sample is weight in two boats – **boat 1 and boat 2**.

1. The sample in **boat 1** is first pyrolyzed in the argon flow. At the preset temperature volatile components of the enclosed carbon compounds escape. In the ideal case and / or after appropriate pretreatment the elementary carbon (EC) remains on the boat.
2. The share of the remaining carbon (EC) in the already pyrolyzed sample is detected by combustion in the oxygen flow.
3. The sample in **boat 2** is converted in the oxygen flow to detect the total carbon content (TC).
4. From the difference of the two measured values (TC and EC) the volume of degradable or active carbon can be deducted.

5.5 Data analysis in the C/S mode

5.5.1 Calibration techniques

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 related to mass. Linear and quadratic calibration functions are calculated in accordance with the equations (5) and (6) through regression calculation.

$$c\ = (k_1 \times I_{net} + k_0)/m \qquad (5)$$

$$c = (k_2 \times I_{Netto}^2 + k_1 \times I_{net} + k_0)/m \qquad (6)$$

c …	target content of the standard
m …	sample mass
I_{net}	net integer
k_0, k_1, k_2 …	calibration coefficient

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.

The multiWin software offers the option to proceed with different calibration strategies adapted to the analytical requirement and dependent on the measuring range and sample matrix. With the multi EA 4000 multi point calibrations with variable sample volumes and constant concentrations are performed.

5.5.2 Day factor

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

The day factor is calculated in accordance with the equation (7).

$$F = \frac{c_{nominal}}{c_{actual}} \qquad (7)$$

5.5.3 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.

5.5.4 Other calculations

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

When measuring TOC with the automatic TIC solids module in the differential method or detecting the carbon after pyrolysis (TC-EC), only a single determination is carried out.

Average value

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

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.

5.6 Cl detection

The samples to be examined enter the combustion chamber in a quartz glass boat via an open gas airlock. The sample is first pyrolized in the inert gas flow (argon). The resulting pyrolysis products are fully incinerated in the oxygen flow after some delay.

The reaction completes in accordance with the following equation:

$$R\text{-}Cl \rightarrow HCl + CO_2 + H_2O + oxides \qquad (8)$$

The share of HCl is determined coulometric after drying the measuring gas. The chloride ions then react completely with the electrolytically generated silver ions to form silver chloride.

$$Ag \rightarrow Ag^+ + e^- \qquad (9)$$

$$Ag^+ + Cl^- \rightarrow AgCl \qquad (10)$$

From the charge volume used up for generating the silver ions the chloride volume can be calculated using Faraday's law.

5.7 Data analysis in the Cl mode

5.7.1 End point routine

After filling the measuring cell or after each change of the electrolyte solution an end point routine must be performed defining the shutdown threshold of the system. The calculated indication value represents both the start point and the end point of titration. The end point routine is started through the multiWin software.

5.7.2 Measuring with split / split factor

In the operating mode with splitting the measuring gas from the combustion chamber is divided in a defined ratio. Only a part of the reaction gas reaches the measuring cell for analysis, thus also permitting the detection of high concentrations.

For Cl content in the low concentration range, no gas splitting is used. The measuring gas passes completely into the drying unit and the measuring cell.

For working with gas splitting, shares of 10 % and 20 % of the measuring gas can be chosen. The actual split factor of a method can be entered/viewed in the method window on the CALIBRATION tab as DAILY FACTOR (menu item METHOD / EDIT).

IMPORTANT

When creating a new split method, the split factor must first be transferred manually from an existing method with the same split ratio.

The actual split factor is checked and, where necessary, corrected using standards.

5.7.3 Analysis

The coulometric Cl detection is an absolute analysis method. No calibration is performed. The result is stated in the analysis report as absolute mass [µg] and as concentration [µg/g] or [mg/g].

6 First commissioning

6.1 Site requirements

6.1.1 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

As far as possible, the atmosphere in the laboratory should be free of TOC and dust and free from draft, corrosive organic 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 walls 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!

6.1.2 Space requirement

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

6.1.3 Energy supply

CAUTION

The analyzer multi EA 4000 must only be connected to a properly grounded mains outlet in accordance with the voltage specifications on the type plate!

The multi EA 4000 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.

6.1.4 Gas supply

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

The connection hoses with outer diameter 6 mm and inner diameter 4 mm are included with the delivery.

6.2 Unpacking and placing the analyzer

IMPORTANT

The analyzer multi EA 4000 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 EA 4000 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.

7 Operation

7.1 Switching on / heat-up phase / setup

CAUTION! CONDENSATE!

If the storage and the installation temperature differ a lot, wait until the multi EA 400 has adapted to the new ambient temperature before connecting it in order to prevent damages to the device by condensation water.

Before switching on check the following:

- ☐ Gases (oxygen and argon (Cl detection or detection of the elementary carbon)) are connected with a system pressure of 4 – 6 bar
- ☐ A combustion tube is inserted into the furnace.
- ☐ The gas supply hoses are connected to the gas airlock.
- ☐ The analysis modules are connected.
- ☐ The sampler (optional) has been installed.
- ☐ The PC is connected and the multiWin software is installed.

Switching on sequence

1. Turn on the gases at the pressure reducer (system pressure 4 – 6 bar).
2. Switch on the PC.
3. Switch on the autosampler FPG 48 (optional).

   ✓ **The autosampler FPG 48 initializes.**
4. Switch on the basic device multi EA 4000.
5. Switch on the analysis module.

   ✓ **The devices will boot. The status lamps at the front panels of the basic device and modules illuminate.**
6. Start the multiWin program.
7. Complete the user login.

   As USER and PASSWORD always enter **Admin**. This user has been configured during the first program installation and has administrator rights.
8. Click on the [INITIALIZE ANALYZER] button.

   ✓ **The initialization and the automatic detection of all connected and switched-on components is performed after successful login.**

Warm-up and run-in phases

After successful login the automatic initialization and query of the components starts. The current values are displayed in the status window:

- ☐ Measuring gas flow: Appears after switching on the suction pump (100 ± 5 l/h).
- ☐ Suction pump in the basic device: The pump switches on 50 K before reaching the target temperature.
- ☐ Furnace temperature: The heating duration is approx. 30 – 40 min. dependent on the specified target temperature.

- NDIR detector (C, S or C/S detection only): This assembly requires a run-in time of approx. 15 min. after switching on the C/S module.
- Coulometer (Cl detection only): run-in time of approx. 10 min.

During this time the indications of the components that are not yet ready for operation are shown in red in the status window.

7.2 C/S measurement

7.2.1 Preparing the multi EA 4000 and C/S module

WARNING! ELECTRIC SHOCK!

Lethal voltages may occur in the combustion furnace. The combustion tube is part of the cover of live components.

Therefore, only switch on the analyzer with the combustion tube inserted! For conversion and maintenance activities at the combustion tube or combustion furnace, always switch off the analyzer from the mains switch and disconnect the multi EA 4000 by removing the mains plug from the mains network.

Check the electric and pneumatic connections in accordance with section "Installing the multi EA 4000 C/S" p. 122 ff.

Fit the ceramic tube to the gas airlock and insert it into the combustion furnace

A ceramic combustion tube is used for the C/S measurements.

Caution: The ceramic tube is chamfered on one side to center it for acceptance in the furnace. The chamfered end is introduced into the furnace. The airlock is attached to the end without chamfer.

1. Fill the chamfered end of the ceramic tube with quartz wool to a thickness of approx. 2.5 cm and push the cotton plug approx. 2 cm into the tube.

   Caution: Do not push the quartz wool too far into the center of the ceramic tube, because the wool clumps up at hot temperatures.

2. Slide the contact pressure ring onto the end of the ceramic tube without chamfer.
3. Slide the sealing ring approx. 3 cm onto the ceramic tube.

	4. Attach the gas airlock until the drill holes of the airlock and the contact pressure ring are on top of each other.
	5. Attach the contact pressure ring to the airlock using three screws. Tighten the screws evenly in turn.
	The gap between the contact pressure ring and the airlock must be approx. 1 mm wide all round.
	6. Push the ceramic tube into the combustion furnace. The gas connection at the airlock should be slightly turned to the left (approx. 45°).
	7. With a slight rotating movement press the gas airlock against the wall holder of the multi EA 4000. The ceramic tube then slides into the receptacle of the combustion furnace. The ceramic tube is positioned correctly if the gas connection points vertically up and the gap between the airlock coupling and the gas airlock is approx. 2 mm wide.

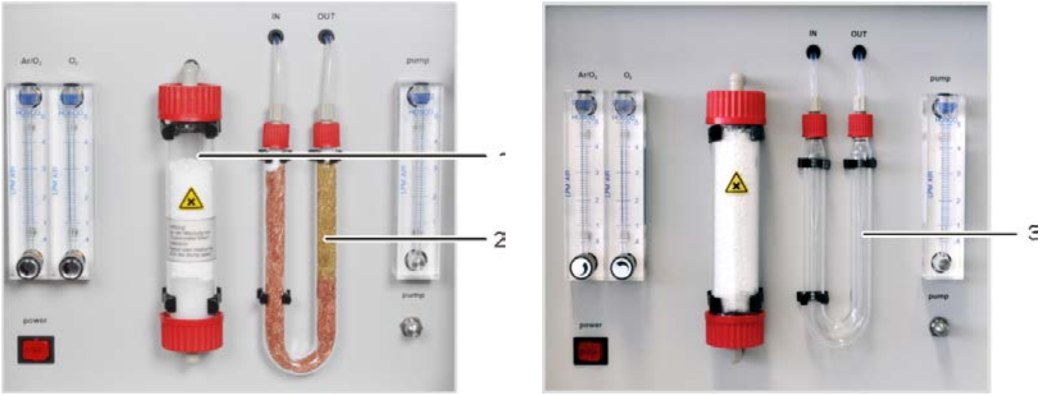
	8. Attach the hose for the oxygen supply to the gas airlock and secure it with the union nut.
	9. To secure the gas hose in a bend **without kinking**, place the hose into the clip on the right-hand side of the analyzer panel and twist the clip.

Checking the halide trap and drying tube

- ☐ Make sure that the drying tube is filled with magnesium perchlorate and quartz wool is only present in the lower part of the tube.
- ☐ For C detection install the halide trap filled with copper and brass wool in the gas path.
- ☐ For S detection remove the halide trap and connect an empty U tube instead.

IMPORTANT

The halide trap absorbs sulfur. Therefore, the use of the halide trap must be evaluated for C/S detection. Dependent on the matrix it is recommended for low sulfur concentrations not to use a halide trap, but the halide trap should be installed in the gas path for higher concentrations.



1	drying tube with magnesium perchlorate	3	empty U tube for S detection
2	halide trap with copper and brass wool for C detection		

Fig. 30 **Checking the halide trap and drying tube**

7.2.2 Performing the C/S measurement

1. Switch on the basic device multi EA 4000, the C/S module and, where applicable, the FPG 48 from the main switch.

   ✓ **The status lamps at the device front panels illuminate.**

   Note:
   If an automatic TIC solids module has been integrated, switch this on, too. This causes the longer transport path of the boat due to the intermediate TIC reactor to be taken into account.
   Do not switch on any Cl module present.

2. Start the multiWin program.
3. Change between the different analyzer modules, first activating the sensors:
   - Select the menu item Device / Device - edit.
   - Activate the Elements to be analyzed.
   - If both carbon/sulfur and chlorine have been activated, click on Ceramic tube (CS) in the list Combustion tube.
   - As States enable the option Solid.
4. Then initialize the system by clicking on [Initialize analyzer].
5. Load a method (menu item Method / Method - activate) or create a new method (menu item Method / Method- new).

   You can select the following parameters for the C/S measurements:
   - TC (only detect total carbon content)
   - TS (only detect total sulfur content)
   - TC+TS (detect total carbon content and total sulfur content)
6. After the run-in time set the following gas flows:

pump	O_2	Ar/O_2
set such that in the window Status analyzer the value target = 100 ± 5 is displayed corresponds to approx. 1.7 l/min	2.3 – 2.5 l/min	-

   The analysis system now has time for conditioning.

   If the intake flow differs greatly or changes subsequently, re-adjust it at the "pump" valve of the multi EA 4000.

7. Weigh the samples on ceramic boats.
8. Start the measurement by clicking on [Start measurement].
9. Create or select an analysis group for storing the measuring data.
10. Create an analysis sequence.
    - Add the desired number of samples to the sample table with ⊞.
    - Enter the following data:

Column	Description
Sampling position	*only for measurements with the autosampler.* Position of the sample on the sample rack. The sample rack assignment may only take place with ascending position numbers. However, it need not be without gaps.

NAME	Sample ID With an automatic sample ID can be generated.
ANALYSIS TYPE	Selection of the analysis type (SAMPLE, DAILY FACTOR, CALIBRATION MEASUREMENT, AQA MEASUREMENT or BOAT BLANK DETERMINATION)
NOMINAL VALUE	Target concentration for calibration standards or AQS standard
Operands	Entry of the weighted sample

- Populate the sampler. Use the buttons and to move the sample rack into the desired position.
- Click on in the sequence row to release it for analysis. All sequence rows can be released by clicking on .

✓ **Sequence rows released for analysis have a green fill.**

11. Click on [START MEASUREMENT].

    Note:
    During measurements with manual sample feed an additional input window appears before each measurement start to enter the weighted sample.

    ✓ **The analyzer starts processing the analysis sequence. The MEASUREMENT - ANALYSIS window opens.**

IMPORTANT

During prolonged pauses during measurement the oxygen supply can be switched off in the multiWin program saving oxygen accordingly. However the oxygen supply must be available again in time (approx. 10 min) before starting the measurement.

7.3 Carbon detection after pyrolysis

7.3.1 Preparing the multi EA 4000 and C/S module

WARNING! ELECTRIC SHOCK!

Lethal voltages may occur in the combustion furnace. The combustion tube is part of the cover of live components.

Therefore, only switch on the analyzer with the combustion tube inserted! For conversion and maintenance activities at the combustion tube or combustion furnace, always switch off the analyzer from the mains switch and disconnect the multi EA 4000 by removing the mains plug from the mains network.

For the detection of carbon after pyrolysis the multi EA 4000 must be equipped with the optional pyrolysis mode.

1. Check the electric and pneumatic connections in accordance with section "Installing the multi EA 4000 C/S" p. 122.Check in particular the argon connection at the multi EA 4000.
2. Attach an exhaust device above the gas airlock on the right-hand side of the EA 4000 to catch and remove the escaping pyrolysis gases.
3. Perform all the other modifications of the analyzer as described in section "Preparing the multi EA 4000 and C/S module" p. 52:
   - Install the ceramic tube with the gas airlock in the combustion furnace.
   - Make sure that the copper and brass wool in the halogen trap are not discolored.
   - Install the autosampler.

7.3.2 Performing the carbon detection after pyrolysis

1. Switch on the basic device multi EA 4000, the C/S module and, where applicable, the FPG 48 from the main switch.

   ✓ **The status lamps at the device front panels illuminate.**

   Note: Do not switch on any Cl module present. The automatic TIC solids module must not be switched on.
2. Start the multiWin program.
3. Change between the different analyzer modules, first activating the sensors:
   - Select the menu item DEVICE / DEVICE - EDIT.
   - Activate the ELEMENTS to be analyzed.
   - If both carbon/sulfur and chlorine have been activated, click on CERAMIC TUBE CS in the list COMBUSTION TUBE.
   - As STATES enable the option SOLID.
4. Then initialize the system by clicking on [INITIALIZE ANALYZER].
5. Load a method (menu item METHOD / METHOD - ACTIVATE) or create a new method (menu item METHOD / METHOD- NEW).
   - Select the parameter "EC/TC" to detect the carbon after pyrolysis.
   - Recommended parameters for the method configuration (tab PROCESS):

FURNACE TEMPERATURE	850 °C
EC PURGE TIME	300 s

6. After the run-in time set the following gas flows:

pump	**O_2**
set such that in the window STATUS ANALYZER the value NOMINAL = 100 ± 5 is displayed corresponds to approx. 1.7 l/min	2.3 – 2.5 l/min

The analysis system now has time for conditioning.

If the intake flow differs greatly or changes subsequently, re-adjust it at the "pump" valve of the multi EA 4000.

7. Weigh the samples on two ceramic boats each.
8. Start the measurement by clicking on [START MEASUREMENT].
9. Create or select an analysis group for storing the measuring data.
10. Create an analysis sequence.

    – Add the desired number of samples to the sample table with . Two boats must be provided for each sample to be processed consecutively in the sequence.

    – Enter the following data:

Column	**Description**
SAMPLE POSITION	Sample position on the sample rack or in the analysis sequence during measurements with manual sample feed The sample rack assignment may only take place with ascending position numbers. However, it need not be without gaps.
NAME	Sample ID With an automatic sample ID can be generated.
ANALYSIS TYPE	Selection of the analysis type (SAMPLE, DAILY FACTOR, CALIBRATION MEASUREMENT, AQA MEASUREMENT or BOAT BLANK DETERMINATION)
NOMINAL VALUE	Target concentration for calibration standards or AQA standard
Operands	Entry of the weighted sample

    – Populate the sampler. Use the buttons and to move the sample rack into the desired position.

    – Click on in the sequence row to release it for analysis. All sequence rows can be released with the button .

    ✓ **Sequence rows released for analysis have a green fill.**

11. Click on [START MEASUREMENT].

    Note: During measurements with manual sample feed an additional input window appears before each measurement start to enter the weighted sample.

    ✓ **The analyzer starts processing the analysis sequence. The MEASUREMENT - ANALYSIS window opens.**

- In the sample in boat 1 the elementary carbon (EC) is determined. Before measuring **boat 1** the suction pump is switched off automatically and a change from oxygen to argon is made. Now set the argon flow at the **Ar/O_2** rotameter to 2.3 – 2.5 l/min. This flow configuration remains in place for subsequent pyrolysis measurements.
- The sample in **boat 1** is first pyrolized in the argon flow after expiry of the waiting time (flushing the combustion tube with argon). At the preset temperature volatile components of the enclosed carbon compounds escape. In the ideal case and / or after appropriate pretreatment the elementary carbon (EC) remains on the boat.
- After pyrolysis a change from argon to oxygen is made and the suction pump is switched on automatically. The share of the remaining carbon (EC) in the already pyrolyzed sample is detected by combustion in the oxygen flow.
- The sample in **boat 2** is converted in the oxygen flow to detect the total carbon content (TC).
- From the difference of the two measured values (TC and EC) the volume of degradable or active carbon can be deducted.

IMPORTANT

During prolonged pauses during measurement the gas supply can be switched off in the multiWin program saving oxygen accordingly. However the oxygen supply must be available again in time (approx. 10 min) before starting the measurement.

7.4 Cl detection

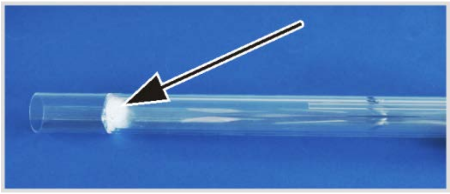
7.4.1 Preparing the multi EA 4000 for Cl measurements

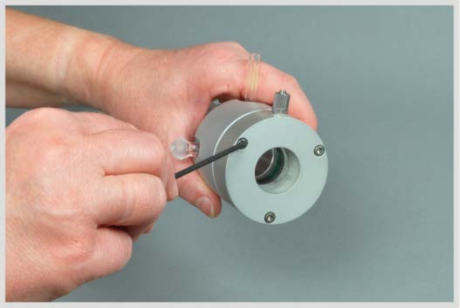
WARNING! ELECTRIC SHOCK!

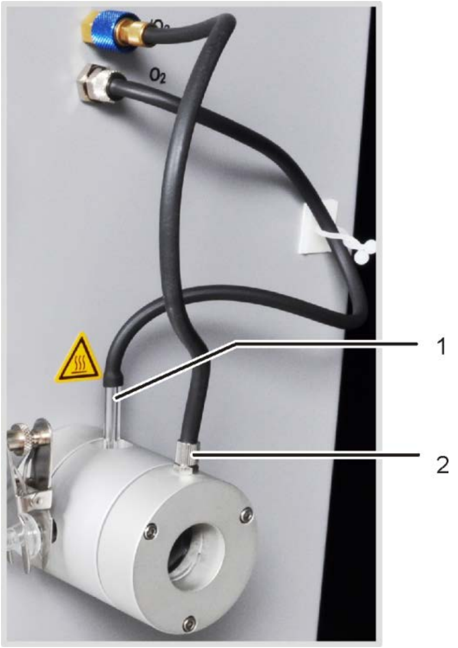
Lethal voltages may occur in the combustion furnace. The combustion tube is part of the cover of live components.

Therefore, only switch on the analyzer with the combustion tube inserted! For conversion and maintenance activities at the combustion tube or combustion furnace, always switch off the analyzer from the mains switch and disconnect the multi EA 4000 by removing the mains plug from the mains network.

Insert the quartz tube into the combustion furnace

	1. Fill the quartz tube with approx. 2.5 cm quartz wool and push the cotton plug into the end of the tube.

	2. Place the sealing ring into the contact pressure ring of the gas airlock.
	3. Slide the quartz tube into the contact pressure ring until the gas outlets protrude into the slots of the ring.
	4. Attach the counter piece of the gas airlock with the three corresponding hexagon socket screws. Tighten the screws evenly in turn. **Note**: When tightening the screws make sure that the quartz tube is centered in the gas airlock.
	✓ **The gas airlock is fitted correctly if the gap between the contact pressure ring and the counter piece is approx. 1 mm wide all round.**



1	oxygen connection
2	argon connection

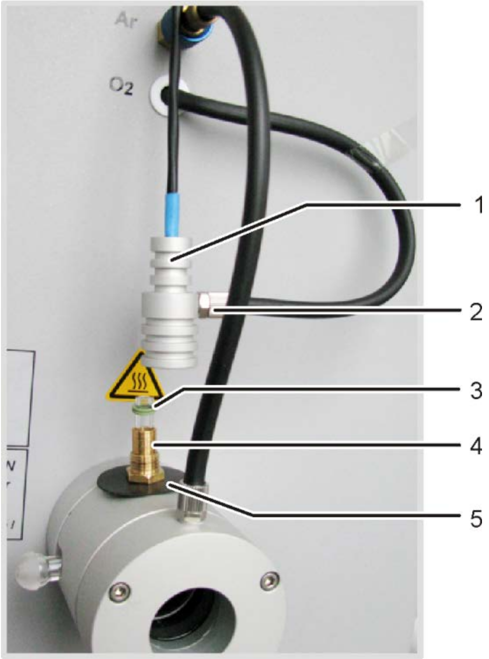
5. Slide the quartz tube into the combustion furnace up to the stop in the coupling of the analyzer panel.
6. *Without using the flame sensor*:
   Slide the hose for the oxygen supply onto the gas connection of the combustion tube (1).
7. Connect the hose for the argon supply to the gas airlock (2).

   ✓ **The combustion tube is inserted into the multi EA 4000.**

Caution:

The hoses must not be kinked or twisted!

Connecting the flame sensor (optional)



1	sensor head
2	oxygen gas connection
3	sealing rings
4	brass sleeve with screw thread
5	aperture disk

1. Connect the hose for the oxygen supply (2) to the sensor head (1).
2. In turn slide the black aperture disk (5), the brass sleeve and the two sealing rings onto the gas connection of the combustion tube in accordance with the figure.

	3. Place the sensor head onto the gas connection and secure it with the brass sleeve. The sealing rings are pressed into the sensor head and seal the flame sensor. 4. Connect the flame sensor connection between the connection of the multi EA 4000 with the C/S module (or Cl module) at the "valve" connection of the multi EA 4000.

Connecting the sulfuric acid container

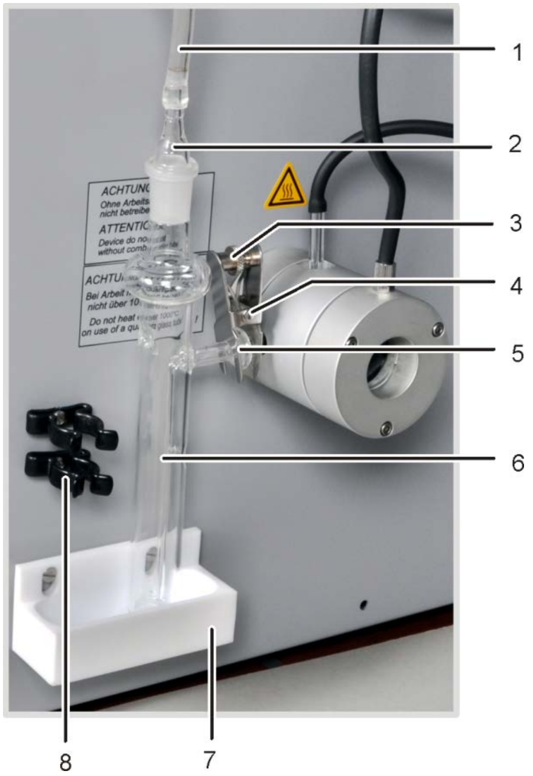
WARNING

The concentrated sulfuric acid used as drying agent in the sulfuric acid container can cause severe chemical burns!

Wear appropriate protective clothing when changing the sulfuric acid! Observe all requirements in the EC safety data sheet! Fill the sulfuric acid with great care!

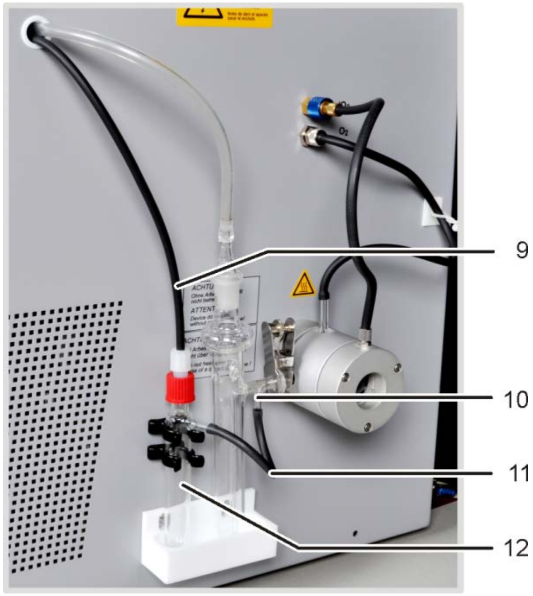
Dependent on the measuring method, a sulfuric acid container with or without splitting outlet is used.

	1. Push the measuring gas hose (colorless) and the bypass hose (black) through the duct in the multi EA 4000. If necessary, use tweezers to pull the hoses out at the other duct end. Feed the hoses through the opening on the right-hand side of the Cl module.



1	measuring gas hose	5	spherical joint
2	glass olive	6	sulfuric acid container
3	Knurled head screw	7	PTFE tray
4	fork clamp	8	clamps for safety container

2. Fill the sulfuric acid container with 12 – 15 ml sulfuric acid.
3. Hold the filled sulfuric acid container above the PTFE tray (7) next to the inlet to the combustion tube at the basic device.
4. Combine the gas outlet of the combustion tube and the inlet of the sulfuric acid container using the spherical joint (5).
5. Secure the spherical joint with the fork clamp (4) and tighten the knurled head screw (3) finger-tight.
6. Seal the sulfuric acid container with the gas olive with hose connection.
7. Push the measuring hose (transparent) onto the gas olive connection.



9	bypass hose	11	connection hose
10	splitting outlet	12	safety container

Sulfuric acid containers with splitting outlet only

8. Connect the splitting outlet of the sulfuric acid container to the side connection of the safety container.
9. Press the safety container into its holder.
10. Connect the black bypass hose (9) at the top of the safety container (12).

    Note: For sulfuric acid containers without splitting outlet the connection hose hangs down loosely.

Sulfuric acid containers with splitting outlet only

11. Connect the bypass hose to the gas inlet branch of the halide trap filled with copper wool.

7.4.2 Preparing the electrolyte solution

WARNING

The 100% acetic and nitric acid used to prepare the electrolyte solution can cause severe chemical burns!

Wear the appropriate protective clothing when preparing the electrolyte solution!

Observe all requirements in the EC safety data sheets!

Required reagents:

200 ml	acetic acid = 99 %
4 ml	concentrated nitric acid
4 g	gelatin
1,0 g	thymol
0.3 g	thymol blue
500 ml	methanol

Prepare the electrolyte solution as follows:

1. solution A:

   fill 500 ml water into a 1000 ml measuring flask, add 4 ml HNO_3 (conc.), carefully add 200 ml acetic acid and fill with water up to the marking.

2. solution B1:

   mix 4 g gelatin in a beaker with 400 ml, allow to swell for 3 hours and then dissolve whilst heating to (35 – 45) °C.

3. solution B2:

   dissolve 1.0 g thymol and 0.3 g thymol blue in a beaker in 500 ml methanol.

4. solution B:

After cooling down to (18 – 22) °C slowly add solution B1 to solution B2 whilst mixing, filter, transfer to a 1000 ml measuring flask and fill with water up to the marking.

5. solution C – ready electrolyte:

   pipette 8 ml of solution B into a 100 ml measuring cylinder and fill with solution A to 100 ml or
   pipette 40 ml of solution B into a 500 ml measuring cylinder and fill with solution A to 500 ml.

Storage and durability of the electrolyte solutions

- Solutions A and B last approx. 6 months if stored in tightly sealed bottles at 4 ± 3 °C.
- The ready electrolyte solution (solution C) lasts approx. 30 days in tightly sealed glass containers at 20 – 25 °C.

7.4.3 Making the measuring cell available

CAUTION

If the electrolyte dries on the combination electrode this may cause an irreversible damage of the electrode. During a brief pause in operation, for example from one day to the next, store the electrode in the electrolyte solution.

IMPORTANT

Leave the electrode for 1-2 h in electrolyte solution prior to first use. Reproducible values are only possible after that.

The filled measuring cell and the electrode must be connected to the device **before** the Cl module is switched on.

1. Fill the electrolyte into the basic body of measuring cell (for the preparation of the electrolyte see section "Preparing the electrolyte solution" p. 64):
   - "sensitive" cell: approx. 20 ml
   - "high concentration" cell: approx. 120 ml
2. Place a magnetic stirring rod onto the floor of the measuring cell.
3. Attach the measuring cell lid using the 3 knurled head screws.
4. Place the measuring cell into the holder of the Cl module.

1	connection for combination electrode
2	connection between measuring gas and combination electrode
3	olive with connection extraction hose
4	measuring cell connection
5	adsorption tube, filled with active carbon

5. Insert the combination electrode and the hose olive with extraction hose into the openings provided in the measuring cell lid
6. Connect the combination electrode and the measuring cell at the connections at the backplate of the Cl module.
7. Slide the measuring hose (colorless) approx. **1 cm wide** onto the end of the combination electrode.
8. Switch on the multi EA 4000 and its device components (see section "Performing the Cl measurement" p. 67).
9. Perform an end point routine (see section "End point routine" p. 66).

   ✓ **Once the end point routine is completed, the system is ready for measurements.**

10. If necessary, check the cell using a direct method.

7.4.4 End point routine

An end point routine is required after each electrolyte change.

The operating point of the coulometric measuring cell is in the range of 1500 – 5000 counts.

1. Start the end point routine manually via the menu item SYSTEM / END POINT ROUTINE.
2. Pull the olive with the extraction hose out of the measuring cell lid. After the prompt meter the HCl solution directly into the measuring cell:

– "sensitive" cell	100 µl of a 0.01N HCl solution
– "high concentration" cell	100 µl of a 0.1N HCl solution

3. Immediately after metering start the end point routine by clicking on [OK].

   In the STATUS ANALYZER window the status END POINT ROUTINE is shown during the process. After the end point routine the window STATUS ANALYZER shows the status STAND-BY TITRATION and the current indicator value.

   ✓ **The system is ready for measurements.**

The calculated operating point of the combination electrode is displayed under the menu item System / Component test in the tab Cl-AMP.

IMPORTANT

To protect the combination electrode against unnecessary wear, note the following:

- ❑ Always fill fresh electrolyte into the measuring cell before the end point routine.
- ❑ Never perform the end point routine several times consecutively.

7.4.5 Performing the Cl measurement

1. Switch on the basic device multi EA 4000, the Cl module, C/S module (only for multi EA 4000 C/S Cl) and, where applicable, the FPG 48 from the main switch.

   ✓ **The status lamps at the device front panels illuminate.**

2. Start the multiWin program.
3. Change between the different analyzer modules, first activating the sensors:
   - Select the menu item Device / Device edit.
   - Activate the chlorine as Element to be analyzed.
   - If both carbon/sulfur and chlorine have been activated, click on Quartz tube (Cl) in the list Combustion tube.
   - As States enable the option Solid.
4. Then initialize the system by clicking on [Initialize analyzer].
5. Load a method (menu item Method / Method - activate) or create a new method (menu item Method / Method- new).

   You can select the following parameters for the Cl measurements:
   - Cl_AMP (detecting chlorine content in solids)
   - Cl_direct_AMP (check the measuring cell by pipetting a liquid standard directly into the measuring cell)
6. Adjust the gas flows:

   Cl measurements without gas splitting
   - For the non-splitting operation Split 100 % must be selected in the method in the tab Process.
   - The following gas flows must be set for that purpose:

Gas flows for measurements without gas splitting		
pump	**O_2**	**Ar**
approx. 600 ml/min	300- 400 ml/min	300- 400 ml/min

   - The following must be noted when adjusting the gas flows:

     The intake flow must be approx. 200 ml/min greater than the oxygen flow.

     To guarantee the effect of the gas airlock the sum of argon and oxygen flow must be at least 200 ml/min greater than the oxygen flow.

     When prompted the user must reduce the oxygen flow manually at the "pump" flow meter of the basic device.

CAUTION

It is essential to reduce the oxygen flow in the non-splitting operation when prompted! Otherwise sulfuric acid or electrolyte solution can be sucked into the hose system and downstream components and cause damage there.

Cl measurements with gas splitting

– In the method parameters set on of the splitting ratios below on the tab PROCESS:

Measuring gas (in %)	Splitting ratio
10	1 : 10
20	1 : 5

– The following gas flows must be set for working with measuring gas splitting:

Gas flows for measurements with measuring gas splitting		
pump	O_2	Ar
max. (approx. 2.5 l/min)	1 l/min	2.4 l/min

– Initially take the splitting factor from an existing method and then calculate it afresh.

7. Weigh the samples on quartz boats.

   Note:
   Samples, such as waste oil, should be placed on annealed quartz sand and covered with an adequate amount of quartz sand during weighing in the quartz glass boat. Waste oil weighted samples may not exceed 50 mg.

8. Start the measurement by clicking on [START MEASUREMENT].
9. Create or select an analysis group for storing the measuring data.
10. Create an analysis sequence.

– Add the desired number of samples to the sample table with .

– Enter the following data:

Column	Description
SAMPLE POSITION	Position of the sample on the sample rack. The sample rack assignment may only take place with ascending position numbers. However, it need not be without gaps.
NAME	Sample ID With an automatic sample ID can be generated.
ANALYSIS TYPE	Selection of the analysis type (SAMPLE, DAILY FACTOR, AQA MEASUREMENT or BOAT BLANK DETERMINATION)
NOMINAL VALUE	Target concentration for AQA standards
Operands	Entry of the weighted sample

– Populate the sampler. Use the buttons and to move the sample rack into the desired position.

– Click on in the sequence row to release it for analysis. All sequence rows can be released with the button .

✓ **Sequence rows released for analysis have a green fill.**

11. Click on the button [START MEASUREMENT].

    The sample is pyrolized at temperatures between 300 and 600 °C (front furnace zone). The resulting pyrolysis products are completely oxidized in the oxygen flow at 1000 °C.

    Using the autosampler FPG 48

    - Select a suitable program for the feed with the autosampler that includes an extended dwell time in the heating area near the furnace inlet
    - With very reactive samples use the optionally available flame sensor.

    Using manual sample feed

CAUTION

When loading the furnace whilst at the same time monitoring the combustion, protective goggles must be worn.

- Start the measurement before the boat is moved into the furnace.
- Push the boat up to just behind the gas airlock into the combustion tube so that the center of the boat is at the level of the front panel, and allow it to heat up briefly.
- Then push the boat carefully into the hot zone of the combustion tube. Monitor the end of the oxygen supply tube constantly. As soon as there are luminous effects from the combustion of the pyrolysis gases, hold the boat in its current position. Once the luminescence dies down, slowly move the boat on. Stop again when there is luminescence, until the boat is fully in the furnace (the position is predefined by the stop of the feeding tool). In this position the volatile pyrolysis products are incinerated at 1000 – 1100 °C.
- After transferring the boat into the hot zone confirm the on-screen message with [OK].

IMPORTANT

The advance of the boat must be very slow to allow the pyrolysis gases to develop slowly and be incinerated completely. Feeding the sample too fast can result in incomplete combustion and sooting up of the analysis system with incorrect measuring results as a consequence.

✓ **The analyzer starts processing the analysis sequence. The MEASUREMENT - ANALYSIS window opens. The measuring routine with titration delay starts.**

7.4.6 Checking the splitting ratio

The splitting ratio set must be checked before a measuring series with a standard and any deviations must be corrected.

The following standard concentrations are recommended:

Measuring cell	Standard concentration
"high concentration"	1 N HCl
"sensitive"	0.1 N HCl

1. Move 50 µl of the standard on ceramic mats in a boat into the hot furnace.
2. Using the method perform a detection:

- Repeat the measurement of the above-mentioned standard 3 times and determine the average value.
- Calculate the splitting factor F:

$$F = \frac{a_{soll}}{\overline{a_{ist}}}$$

a_{soll} … expected absolute Cl content in µg

$\overline{a_{ist}}$ … average absolute Cl content from the 3 standard measurements

- Enter the calculated factor in the method on tab CALIBRATION in the input field DAILY FACTOR and accept the values with [ACCEPT] for the subsequent measurements.

IMPORTANT

To calculate a new splitting factor the DAILY FACTOR must be set to a value of 1 in the method on the tab CALIBRATION .

7.4.7 Cauterizing the quartz boat

Quartz boats have to be cauterized

☐ before first use

☐ after cleaning out heavy contamination.

To this end perform the measuring routine with empty quartz boats.

7.5 TIC/TOC detection with automatic TIC solids module

7.5.1 Preparing the automatic TIC solids module for the analysis

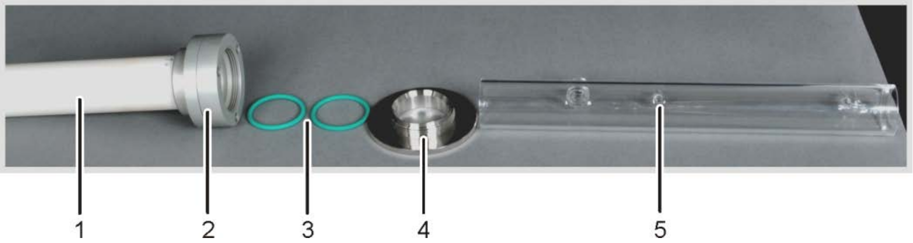
WARNING! ELECTRIC SHOCK!

Lethal voltages may occur in the combustion furnace. The combustion tube is part of the cover of live components.

Therefore, only switch on the analyzer with the combustion tube inserted! For conversion and maintenance activities at the combustion tube or combustion furnace, always switch off the analyzer from the mains switch and disconnect the multi EA 4000 by removing the mains plug from the mains network.

1. Place the automatic TIC solids module to the right of the basic device. Push the TIC solids module up to the wall of the basic device until the spacers touch the device wall.
2. Connect the electrical connections and check the hose connections (see section "Installing the automatic TIC solids module" p. 131).

Attach the TIC reactor to the ceramic tube and insert into the combustion furnace



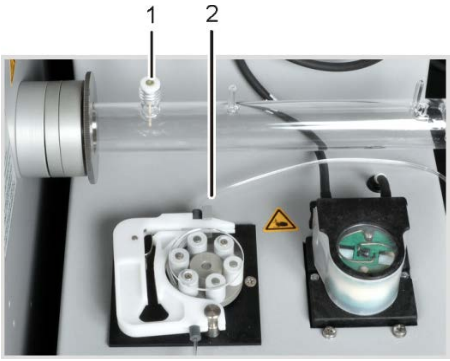
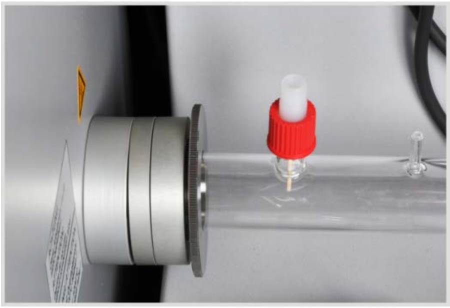
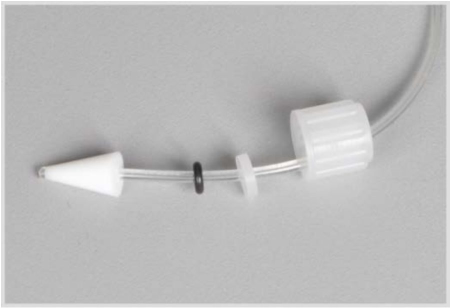
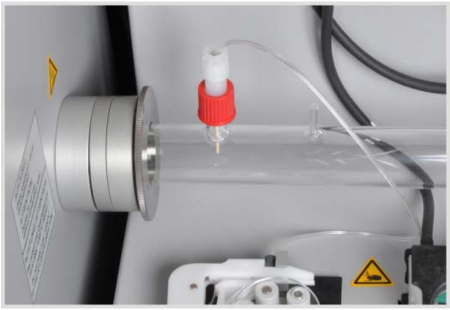
1	ceramic tube	4	clamping ring
2	coupling	5	TIC reactor
3	sealing rings		

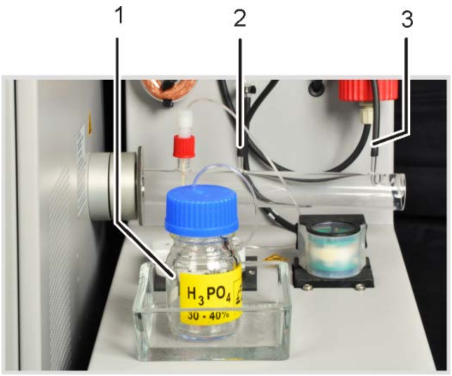
Fig. 31 **Individual components for the connection between the TIC reactor and ceramic tube**

	First attach the coupling with the inside seal to the ceramic tube: 1. Slide the first coupling ring and a sealing ring onto the ceramic tube.

	2. Attach the 2nd ring and secure it with the 3 hexagon socket screws supplied. Tighten the screws in turn. **Caution**: The gap between the two rings should be evenly approx. 1 mm wide.
	3. Place two sealing rings into the coupling.
	4. Screw on the knurled ring with half a rotation.
	5. Slide the reactor into the coupling until it touches the ceramic tube. Attach the TIC reactor by carefully tightening the knurled ring. **Caution**: The reactor must be centered in the holder.
	6. Move the ceramic tube into the opening of the combustion furnace and press it into the rear seal. The reactor connections must point upwards.

Connect the reactor and sampler FPG 48

	1. Place the ceramic cannula (1) onto the port with the screw thread. 2. Clamp the pump hose into the cartridge of the hose pump (2). The hose end with the connection to the reactor must point towards the reactor.
	3. Secure the ceramic cannula with the red screw cap.
 	4. Check that the sealing cone, seals and screw cap are fitted on the pump hose. 5. Screw the pump hose finger-tight to the red screw cap.

<table>
<tr>
<td>
</td>
<td>6. Connect the measuring outlet (2) to the top end of the cooling coil on the rear of the TIC solids module.
7. Slide the hose for the oxygen supply (3 in Fig. 7 p. 25) onto the reactor connection for the carrier gas (3).
Caution:
Always support the reactor with one hand whilst attaching the hoses. If there is too much pressure on the gas cylinder from one side, the TIC reactor can break.
8. Place the reagent tray with the acid bottle into a drip tray in front of the acid pump.</td>
</tr>
<tr>
<td></td>
<td>9. Slide the white PTFE ring onto the end of the TIC reactor.
10. Place the converted sampler FPG 48 (see section "Fehler! Verweisquelle konnte nicht gefunden werden." p. Fehler! Textmarke nicht definiert.) near the TIC solids module. The TIC reactor with the PTF ring rests on the edge of the sampler.
Align the sampler until the ceramic boat can be moved straight into the reactor and further into the furnace during TOC measurements. A gap of 2 - 4 mm must remain between the TIC reactor and the sampler to prevent tension from the thermal expansion of the ceramic tube.</td>
</tr>
<tr>
<td colspan="2">11. To the right of the FPG 48 below the ejection rail place a container for the used ceramic boats.</td>
</tr>
</table>

7.5.2 Required reagents

The following reagents are required for the TIC detection:

- ☐ 40% ortho-phosphoric acid (for recommended differential method)
- ☐ 10% hydrochloric acid for TOC detection (for direct method)

7.5.3 Performing the TIC detection with the automatic TIC solids module

1. In the acid bottle of the TIC solids module keep ortho-phosphoric acid ready.
2. Switch on the basic device multi EA 4000, the C/S module, the automatic TIC solids module and the FPG 48 from the main switch.

   ✓ **The status lamps at the device front panels illuminate.**

   Note: Do not switch on any Cl module present.
3. Start the multiWin program.

4. Change between the different analyzer modules, first activating the sensors:
   - Select the menu item DEVICE / DEVICE - EDIT.
   - Activate "C" in the list ELEMENTS.
   - In the list COMBUSTION TUBE click on CERAMIC TUBE (CS).
   - As STATES enable the option SOLID.
   - Activate the EJECTION RAIL option.
5. Initialize the system by clicking on [INITIALIZE ANALYZER].
6. Load a method (menu item METHOD / METHOD - ACTIVATE) or create a new method (menu item METHOD / METHOD NEW).

   Select the parameter TIC (detect TIC content only).
7. After the run-in time set the following gas flows:

pump	**O_2**	**Ar/O_2***
approx. 1.7 l/min corresponds to TARGET = 100 ± 5 in the STATUS window	1 l/min	1.5 l/min

   ***Caution**:
   Argon is not required for this mode, oxygen is automatically present at the rotameter "Ar/O_2".

   The analysis system now has time for conditioning.

   If the intake flow differs greatly or changes subsequently, re-adjust it at the "pump" valve of the multi EA 4000.
8. Weigh the sample on ceramic boat.
9. Start the measurement by clicking on [START MEASUREMENT].
10. Create or select an analysis group for storing the measuring data.
11. Create an analysis sequence.
    - Add the desired number of samples to the sample table with .
    - Enter the following data:

Column	**Description**
SAMPLE POSITION	Position of the sample on the sample rack. The sample rack assignment may only take place with ascending position numbers. However, it need not be without gaps.
NAME	Sample ID With an automatic sample ID can be generated.
ANALYSIS TYPE	Selection of the analysis type (SAMPLE, DAILY FACTOR, CALIBRATION MEASUREMENT, AQ A MEASUREMENT or BOAT BLANK DETERMINATION)
NOMINAL VALUE	Target concentration for calibration standards or AQA standard
Operands	Entry of the weighted sample

    - Populate the sampler. Use the buttons and to move the sample rack into the desired position.

- Click on ✎ in the sequence row to release it for analysis. All sequence rows can be released with the button ☑.

✓ **Sequence rows released for analysis have a green fill.**

12. Click on [START MEASUREMENT].

    Note: During measurements with manual sample feed an additional input window appears before each measurement start to enter the weighted sample.

    ✓ **The analyzer starts processing the analysis sequence. The MEASUREMENT - ANALYSIS window opens.**

    - The boat pick-up takes a boat from the sample rack and places it onto the ceramic hook.
    - The ceramic hook moves the boat into the TIC reactor. As soon as the boat has reached the position below the ceramic cannula, the acid is added automatically. During the slow boat advance the entire sample is mixed with acid.
    - The measuring gas with the purged TIC is passed via the measuring gas drying and cleaning of the automatic TIC solids module to the detector in the C/S module. By adding the oxygen at the inlet of the TIC reactor the measuring gas is prevented from escaping through the open inlet.

7.5.4 Performing the TOC detection in accordance with the differential method using the automatic TIC solids module (recommended method)

1. In the acid bottle of the TIC solids module keep 40% ortho-phosphoric acid ready.
2. Switch on the basic device multi EA 4000, the C/S module, the automatic TIC solids module and the FPG 48 from the main switch.

   ✓ **The status lamps at the device front panels illuminate.**

   Note: Do not switch on any Cl module present.
3. Start the multiWin program.
4. Change between the different analyzer modules, first activating the sensors:
   - Select the menu item DEVICE / DEVICE - EDIT.
   - Activate "C" in the list ELEMENTS.
   - In the list COMBUSTION TUBE click on CERAMIC TUBE (CS).
   - As STATES enable the option SOLID.
   - Activate the EJECTION RAIL option.
5. Then initialize the system by clicking on [INITIALIZE ANALYZER].
6. Load a method (menu item METHOD / METHOD - ACTIVATE) or create a new method (menu item METHOD / METHOD- NEW).

   Select the TOC parameter.
7. After the run-in time set the following gas flows:

pump	O_2	Ar/O_2
approx. 1.7 l/min corresponds to TARGET = 100 ± 5 in the STATUS window	1 l/min	1.5 l/min

The analysis system now has time for conditioning.

If the intake flow differs greatly or changes subsequently, re-adjust it at the "pump" valve of the multi EA 4000.

8. Weigh the sample on 2 ceramic boats.

   Caution:
   The weighted sample on boat 1 for the TIC detection must be max. 50 mg. Cover the sample with some annealed quartz sand. The quartz sand improves the wetting of the sample with acid.

9. Start the measurement by clicking on [START MEASUREMENT].
10. Create or select an analysis group for storing the measuring data.
11. Create an analysis sequence.

   – Add the desired number of samples to the sample table with ⊞.

   – Enter the following data:

Column	Description
SAMPLE POSITION	Position of the sample on the sample rack. The sample rack assignment may only take place with ascending position numbers. However, it need not be without gaps.
NAME	Sample ID With ✎ an automatic sample ID can be generated.
ANALYSIS TYPE	Selection of the analysis type (SAMPLE, DAILY FACTOR, CALIBRATION MEASUREMENT, AQA MEASUREMENT or BOAT BLANK DETERMINATION)
NOMINAL VALUE	Target concentration for calibration standards or AQA standard
OPERANDS	Entry of the weighted sample

   – Populate the sampler. Use the buttons ↻ and ↷ to move the sample rack into the desired position.

   – Click on ✎ in the sequence row to release it for analysis. All sequence rows can be released with the button ✓.

   ✓ **Sequence rows released for analysis have a green fill.**

12. Click on [START MEASUREMENT].

   Note:

   During measurements with manual sample feed an additional input window appears before each measurement start to enter the weighted sample.

   ✓ **The analyzer starts processing the analysis sequence. The MEASUREMENT - ANALYSIS window opens.**

   – The boat pick-up takes boat 1 from the sample rack and places it onto the ceramic hook.

   – The ceramic hook moves the boat into the TIC reactor. As soon as the boat has reached the position below the ceramic cannula, the acid is added automatically. During the slow boat advance the entire sample is mixed with acid.

   – The measuring gas with the purged TIC is passed via the measuring gas drying and cleaning of the automatic TIC solids module to the detector in the C/S module.

   – Boat 1 is then pulled out of the TIC reactor and deposited on the ejection rail.

- The sampler takes boat 2 with the identical sample from the sample rack and moves it directly into the hot zone of the furnace. Here it is incinerated in the oxygen flow. The total carbon content (TC) is determined in this way.
- The difference of the two measured values (TC-TIC) is then calculated and issued as TOC result in the program.

IMPORTANT

During prolonged pauses during measurement the gas supply can be reduced in the multiWin program saving gas accordingly. However the gas supply must be available again in time (approx. 10 min) before starting the measurement.

7.5.5 TOC measurement with direct detection

CAUTION

Only use HCl acid to purge the TIC from the sample. Phosphoric acid is not suited for the direct detection of the TOC.

Perform a measurement as follows:

1. In the acid bottle of the TIC solids module keep 10% hydrochloric acid ready.
2. Switch on the basic device multi EA 4000, the C/S module, the automatic TIC solids module and the FPG 48 from the main switch.

   ✓ **The status lamps at the device front panels illuminate.**

   Note: Do not switch on any Cl module present.
3. Start the multiWin program.
4. Change between the different analyzer modules, first activating the sensors:
   - Select the menu item DEVICE / DEVICE - EDIT.
   - Activate the ELEMENTS to be analyzed.
   - If both carbon/sulfur and chlorine have been activated, click on CERAMIC TUBE (CS) in the list COMBUSTION TUBE.
   - As STATES enable the option SOLID.
   - Activate the EJECTION RAIL option.
5. Then initialize the system by clicking on [INITIALIZE ANALYZER].
6. Load a method (menu item METHOD / METHOD - ACTIVATE) or create a new method (menu item METHOD / METHOD NEW).

   Select the IC/OC parameter.
7. After the run-in time set the following gas flows:

pump	O_2	Ar/O_2
approx. 1.7 l/min corresponds to TARGET = 100 ± 5 in the STATUS window	1 l/min	1.5 l/min

   The analysis system now has time for conditioning.

   If the intake flow differs greatly or changes subsequently, re-adjust it at the "pump" valve of the multi EA 4000.

8. Weigh the sample on ceramic boat.
9. Start the measurement by clicking on [START MEASUREMENT].
10. Create or select an analysis group for storing the measuring data.
11. Create an analysis sequence.
    - Add the desired number of samples to the sample table with .
    - Enter the following data:

Column	**Description**
SAMPLE POSITION	Position of the sample on the sample rack. The sample rack assignment may only take place with ascending position numbers. However, it need not be without gaps.
NAME	Sample ID With an automatic sample ID can be generated.
ANALYSIS TYPE	Selection of the analysis type (SAMPLE, DAILY FACTOR, CALIBRATION MEASUREMENT, AQA MEASUREMENT or BOAT BLANK DETERMINATION)
NOMINAL VALUE	Target concentration for calibration standards or AQS standard
Operands	Entry of the weighted sample

- Populate the sampler. Use the buttons and to move the sample rack into the desired position.
- Click on in the sequence row to release it for analysis. All sequence rows can be released with the button .

✓ **Sequence rows released for analysis have a green fill.**

12. Click on [START MEASUREMENT].

Note: During measurements with manual sample feed an additional input window appears before each measurement start to enter the weighted sample.

✓ **The analyzer starts processing the analysis sequence. The MEASUREMENT - ANALYSIS window opens.**

- The boat pick-up takes a boat from the sample rack and places it onto the ceramic hook.
- The ceramic hook moves the boat into the TIC reactor. As soon as the boat has reached the position below the ceramic cannula, the acid is added automatically. During the slow boat advance the entire sample is mixed with acid.
- The measuring gas with the purged TIC is passed via the measuring gas drying and cleaning of the automatic TIC solids module to the detector in the C/S module.
- The same boat is then moved further into the furnace to detect the TOC (after removing the TIC) by incineration in the oxygen flow.

7.6 TIC detection with the manual TIC solids module

7.6.1 Prepare the manual TIC solids module and analyzer

The manual TIC solids module only works in conjunction with the C/S module. Here, the measuring gas is not pumped through the detector with the pump of the basic device, but purged in the sealed system with acid and passed with the carrier gas (oxygen) to the detector.

1. Disconnect the connection "pump" and "analyte" between the multi EA 4000 and the C/S module. (All other connection can remain connected).
2. Check the hoses in accordance with the hose diagram (see Fig. 42 p. 135 and Fig. 43 p. 136).
3. Connect the manual TIC solids module to the C/S module:

C/S module connections		**Target**
"PC"	➪	USB connection for PC connection
"scale"	➪	external scales (optional)
socket for mains connector	➪	mains connection
"analyte" connection	➪	TIC module "analyte"
additional connections of the TIC module		
"O_2"	➪	oxygen gas connection with quick-release coupling Set oxygen system pressure to 4– 6 bar.
socket for mains connector	➪	mains connection

CAUTION

Always disconnect the "pump" connection between the C/S module and the multi EA 4000. Otherwise phosphoric acid might be sucked through the system and the detectors might be damaged.

For C/S measurements the "pump" connection must be restored.

7.6.2 Performing the TIC measurement

IMPORTANT

Only use dry 50 ml Erlenmeyer flasks with a standard boat NS 29/32 for the analyses.

The sample must be ground as finely as possible and homogeneous.

In addition to the safety notices for the analyzer multi EA 4000 in section "Safety instructions" p. 11 the following notes apply in particular to the operation of the manual solids module:

WARNING FOR HANDLING PHOSPHORIC ACID!

The reactor container and hoses of the acid supply contain phosphoric acid! Phosphoric acid is irritating for eyes, skin and the mucous membrane!

Meter slowly with the dispenser to prevent excess pressure!

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

CAUTION! OPERATION OF THE MAGNETIC STIRRER!

- Do not operate the heating plate unsupervised!
- Avoid overheating the acid bath! Limit the maximum temperature to 80 °C.
- Place the glass flask in the center of the heating plate to prevent the magnet from knocking against the flask wall.
- Do not set the stirring speed of the magnet too high.

1. Fill the acid bottle with 40 – 50% phosphoric acid and place it into the plastic insert in the upper housing.
2. Screw the dispenser onto the bottle. Connect the acid supply hose to the valve and the TIC head.
3. Switch on the basic device multi EA 4000, the C/S module and the manual TIC solids module from the respective main switch.

   ✓ **The status lamps at the device front panels illuminate.**
4. Switch on the heating plate.

   Adjust the temperature with the left-hand rotary switch of the heating plate. A temperature setting of approx. 80 °C is recommended. If the temperature selection is too high, too much water evaporates, at too low a temperature the reaction takes longer.
5. Start the multiWin program.
6. Change between the different analyzer modules, first activating the sensors:
   - Select the menu item Device / Device - edit.
   - Activate the Elements to be analyzed.
   - In the list Combustion tube click on the option Ceramic tube (CS).
   - As States enable the option Solid.
   - Activate the TIC manual option.
7. Then initialize the system by clicking on [Initialize analyzer].
8. Load a method (menu item Method / Method - activate) or create a new method (menu item Method / Method- new) with the TIC parameter (detect only TIC content).

   Note:
   In the method on the tab Process set the furnace temperature to a value of 0°C. The combustion furnace is then not monitored and the analyzer released for the measurement.
9. At the valve set the oxygen flow to 16 l/h.
10. Weigh the finely ground sample into the Erlenmeyer flask.

    Note: The sample must not stick to the flask wall during transfer. Only the floor of the Erlenmeyer flask is covered with acid. Sample sticking to the walls is lost for analysis.
11. Carefully place the magnetic stirring rod into the Erlenmeyer flask and place the flask into the center of the heating plate.

12. Place the TIC head onto the flask. Rotate the flask and TIC head and press both slightly against each other. The boat connection must be gas-tight to prevent measuring gas from escaping.
13. Start the measurement by clicking on [START MEASUREMENT].
14. Create or select an analysis group for storing the measuring data.
15. Create an analysis sequence.
    - Add the desired number of samples to the sample table with .
    - Enter the following data:

Column	**Description**
NAME	Sample ID With an automatic sample ID can be generated.
ANALYSIS TYPE	Selection of the analysis type (SAMPLE, DAILY FACTOR, CALIBRATION MEASUREMENT, AQA MEASUREMENT or BOAT BLANK DETERMINATION)
NOMINAL VALUE	Target concentration for calibration standards or AQA standard
OPERANDS	Entry of the weighted sample

    - Click on in the sequence row to release it for analysis. All sequence rows can be released with the button .

    ✓ **Sequence rows released for analysis have a green fill.**

16. Click on [START MEASUREMENT].
17. Enter the weighted sample in [mg].
18. Wait for the ready message of the analyzer.
    Before the measurement the analyzer must have been conditioned with oxygen. To this end the air must have been forced out of the flask and hose system and only oxygen must reach the detector. Dependent on the flask size this can take up to 2 min.
19. After the message "PLEASE PRESS 'OK' TO START INTEGRATION AND THEN ADD ACID!." click on [OK].
    Initially meter 2 ml phosphoric acid.
    Slowly set the stirrer to stage 5. The sample must not be flung against the flask wall. Jerky or rapid movements of the stirrer should therefore be avoided at all times.
    Add 2 ml acid each evenly 1 - 3 times (dependent on the concentration and sample volume).

IMPORTANT

Since the release of the CO_2 also depends on the metering speed and the stirring speed of the stirring magnet, always keep to the same cycle and the same speeds for each measurement.

✓ **The analyzer starts processing the analysis sequence. The MEASUREMENT ANALYSIS window opens.**

20. After completing the measurement switch off the stirring magnet and open the flask: Release the TIC head with a rotary movement. Move the tripod arm upwards, tilt it back and lock it in this position.
21. Clean and dry the flask.

22. During the measuring pause connect a dry Erlenmeyer flask to the TIC head. This retains the oxygen flushing (conditioning) and the dry gas flow cleans the hose system of any residual moisture.

23. Keep the heating plate at a constant temperature. Only switch off the temperature control during prolonged measuring pauses.

8 Maintenance and care

Maintenance and care of the analyzer are described in this chapter for the individual modules. Observe all notices for the modules combined in your system.

8.1 Basic device multi EA 4000

Maintenance intervals

Maintenance task	Maintenance interval
clean and maintain the device	weekly
check fastening screws for tight fit	monthly
check all hose connections for tight fit	weekly

8.1.1 Clean and maintain the device

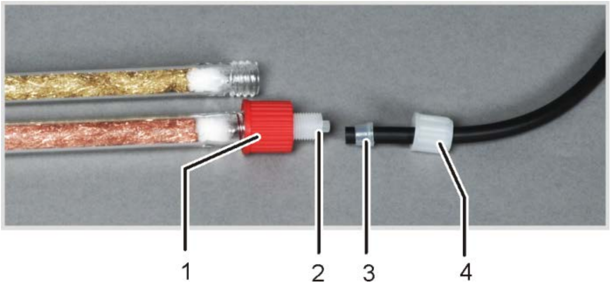
- ☐ Remove chemical splashes (acid, electrolyte solution) immediately with cellulose.
- ☐ Clean the device with a moist cloth.

IMPORTANT

Do not wet the safety notices at the device with solvents. Replace damaged safety notices.

8.1.2 Check the hose connections

To connect hoses to the individual components (halogen trap, drying tube, adsorption tube etc.) Swagelok screw connections of different sizes and materials are mainly used.



1	union nut
2	main screw connection body
3	conical seal
4	union nut

Fig. 32 **Hose connection at the halogen trap**

- ☐ The conical side of the sealing cone points towards the hose end.
- ☐ Only tighten the screw connections manually without tools.
- ☐ Only place the union nuts straight onto the thread without twisting.
- ☐ Make sure that flexible hoses are not twisted or kinked.

8.2 Operation with C/S module

Maintenance intervals

Maintenance task	Maintenance interval
Check ceramic tube for cracks and visible damage, replace	every three months
Check and clean dust trap	monthly (dependent on the incineration and sample matrix)
Replace desiccant in the drying tube	as required (blackening or clumping due to moisture absorption)
Replace halide trap	if half of the copper wool or the brass wool is discolored

8.2.1 Check and replace the ceramic tube

WARNING! ELECTRIC SHOCK!

Lethal voltages may occur in the combustion furnace. The combustion tube is part of the cover of live components.

Therefore, only switch on the analyzer with the combustion tube inserted! For conversion and maintenance activities at the combustion tube or combustion furnace, always switch off the analyzer from the mains switch and disconnect the multi EA 4000 by removing the mains plug from the mains network.

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 (preferably overnight)!

Remove the ceramic tube

1. Arrange to have at least 75 cm of space to the right of the multi EA 4000. To do so, put aside the FPG 48 or manual feeder.
2. Remove the hose for the oxygen supply from the gas airlock.
3. Grip the gas airlock and with a slight rotation pull the combustion tube out of the seal of the dust trap in the rear of the combustion furnace.
4. If the tube moves freely, pull it out straight.
5. Unscrew the hexagon socket screws and dismantle the gas airlock. Slide the sealing rings of the ceramic tube.
6. Remove the quartz wool from the ceramic tube.
7. If the ceramic tube is broken, open the dust trap on the rear of the furnace (see section "Cleaning the dust trap" p. 86) and pull out the tube segment.

   Caution!
   The heating rods of the furnace must not be damaged when removing fragments!

Clean the ceramic tube

1. Blast the dust off the ceramic tube using compressed air. You can remove deposits using a brush.
2. Flush the ceramic tube with ultrapure water and leave to dry completely.

Install the ceramic tube

Install the ceramic tube in accordance with the description in section "Fit the ceramic tube to the gas airlock and insert it into the combustion furnace" p. 52.

8.2.2 Cleaning the dust trap

WARNING! ELECTRIC SHOCK!

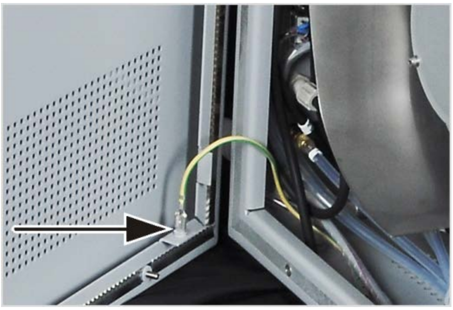
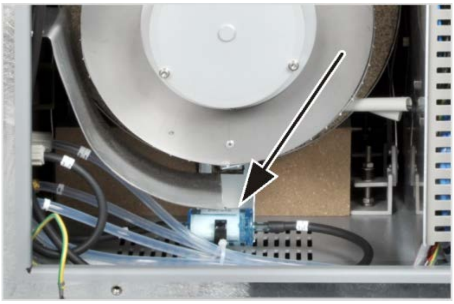
Lethal voltages may occur in the combustion furnace. The combustion tube is part of the cover of live components.

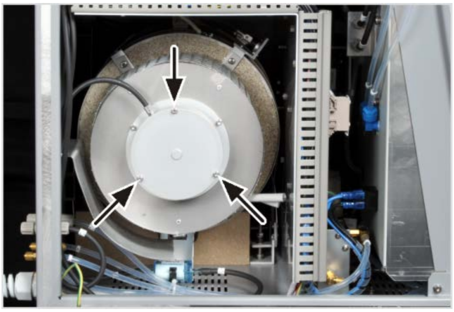
Therefore, only switch on the analyzer with the combustion tube inserted! For conversion and maintenance activities at the combustion tube or combustion furnace, always switch off the analyzer from the mains switch and disconnect the multi EA 4000 by removing the mains plug from the mains network.

CAUTION

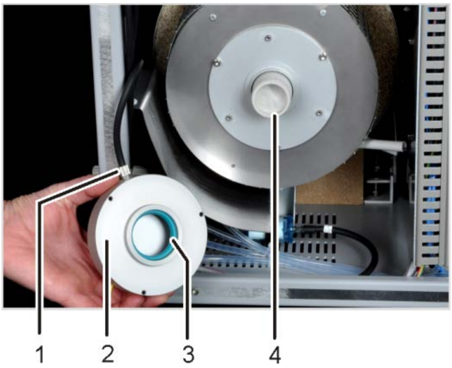
There is a risk of burning on the combustion furnace! Only remove the dust trap when the device is cold or allow the device to cool down sufficiently (preferably overnight)!

An indicator for the cleaning of the dust trap is the dust filter installed downstream of the trap. Check the dust filter monthly for contamination. If contaminated, replace the dust filter and clean the dust trap.

	1. Switch the multi EA 4000 off from the mains switch switch and unplug the mains plug from the mains outlet. 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. Check the dust filter. If it is contaminated, replace it and clean the dust trap as described below. If the dust filter is clean, the dust trap does not need servicing.



4. Unscrew the 3 hexagon socket screws holding the dust trap and remove the dust trap.



1	gas connection
2	dust trap
3	sealing ring
4	ceramic tube with quartz wool

5. Unscrew the union nut at the gas connection and pull off the hose.
   Clean the gas connection.
6. Tap to remove any dust from the dust trap or remove it using a brush.
7. Check the sealing ring and replace it if damaged.
   Observe the installation position of the sealing ring. The closed side must point outwards (towards the user). The open side points to the dust trap.
8. Attach the dust trap again and screw it back gas-tight.

   Caution:
   The measuring gas hose must point to the left again, see figure above.

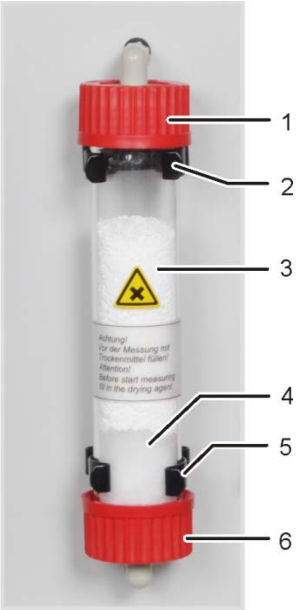
9. Insert the protective conductor into the side wall and attach the side wall.
10. If the ceramic tube has slid out of the airlock coupling on the right-hand side, slide it back in with a slight rotation (see section "Fit the ceramic tube to the gas airlock and insert it into the combustion furnace" p. 52).

8.2.3 Replacing the desiccant in the drying tube

Drying tubes are used in the following measurement arrangements:

- C/S measurement at the basic device multi EA 4000
- TIC measurement in the corresponding TIC solids modules

The desiccant must be replaced if visibly saturated with water If soot or other particles discolor the desiccant, it must be replaced immediately.



1; 6	screw connections
2; 5	retention clips
3	desiccant (magnesium perchlorate)
4	Quartz wool

1. Release the screw connections at the glass tube.

   Caution: The seals must not be lost and must be reinstalled in the same manner.
2. Remove the desiccant and the quartz wool.
3. Flush the glass tube and dry it.
4. Seal the end of the glass tube with quartz wool approx. 2 cm thick.
5. Fill the glass tube with 50 – 60 g fresh desiccant (magnesium perchlorate).
6. Screw the glass tube back tightly and press it back into the holder. Do not kink the hoses.

8.2.4 Replacing the halide trap

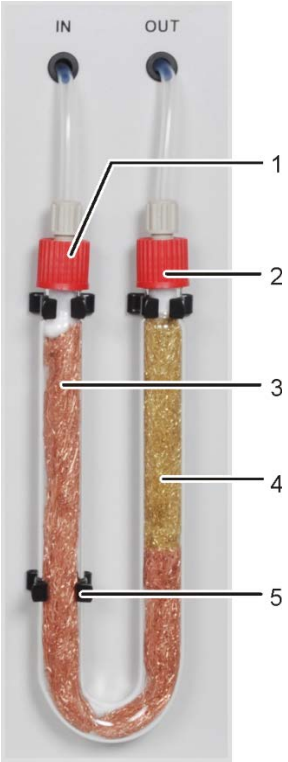
Halide traps are used in the following measurement arrangements:

- TC measurement at the basic device multi EA 4000
- Cl measurement in the Cl module
- TIC measurement in the corresponding TIC solids modules

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 or the brass wool is discolored.



1	gas inlet
2	gas outlet
3	copper wool
4	brass wool
5	clamp

1. Pull the U tube out of the holder.
2. Unscrew the red union nuts from the U tube 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 the gas inlet to the branch with copper wool and the gas outlet to the branch with brass wool.

8.3 Operation with CI module

Maintenance intervals

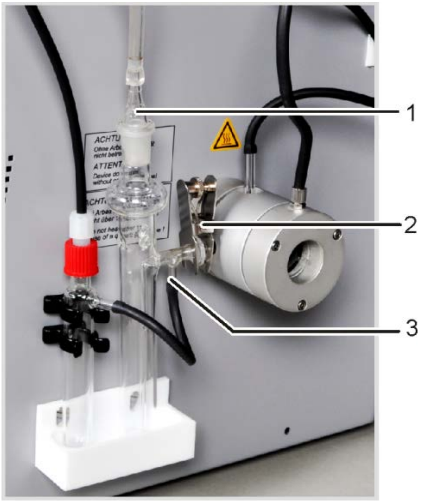
Maintenance task	Maintenance interval
Remove chemical splashes from the housing	immediately
Check combustion tube and replace if necessary	after approx. 100 operating hours
Clean measuring cell	at least weekly, recommendation: wipe clean during electrolyte change
Change electrolyte solution	as required, min. 1x each measuring day
Change the sulphuric acid	if the sulfuric acid drops below 70 %, i.e. if the acid volume increases by 1/3rd
Replace the halide trap	if half of the copper wool or the brass wool is discolored
Replace the adsorption agent	weekly (every 40 operating hours)

Replace the copper and brass wool in the halide trap analog to the replacement of the halide trap in the basic device in the multi EA 4000 (→ see section "Replacing the halide trap" p. 89).

8.3.1 Replacing the sulfuric acid/cleaning the sulfuric acid container

WARNING

The concentrated sulfuric acid used as drying agent in the sulfuric acid container can cause severe chemical burns!
Wear appropriate protective clothing when working at the sulfuric acid container.



1. Pull the glass olive (1) out of the sulfuric acid container.
2. When using a container with splitting outlet: Pull the black hose (3) off the bypass of the sulfuric acid container.
3. Remove the fork clamp (2) and disconnect the spherical joint.
4. Remove the sulfuric acid container carefully from the drip tray.
5. Carefully pour out the sulfuric acid. observe the regulations on the handling of concentrated sulfuric acid.

6. Flush the sulfuric acid container with a small amount of fresh sulfuric acid.
   In case of heavy contamination flush it several times with distilled water and then allow it to dry completely.
7. Fill the sulfuric acid container with approx. 15 ml concentrated sulfuric acid and connect it in accordance with section "Connecting the sulfuric acid container" p. 62.

8.3.2 Servicing/replacing the quartz glass combustion tube

WARNING! ELECTRIC SHOCK!

Lethal voltages may occur in the combustion furnace. The combustion tube is part of the cover of live components.

Therefore, only switch on the analyzer with the combustion tube inserted! For conversion and maintenance activities at the combustion tube or combustion furnace, always switch off the analyzer from the mains switch and disconnect the multi EA 4000 by removing the mains plug from the mains network.

WARNING

The concentrated sulfuric acid used as drying agent in the sulfuric acid container can cause severe chemical burns!
Wear appropriate protective clothing when working at the sulfuric acid container.

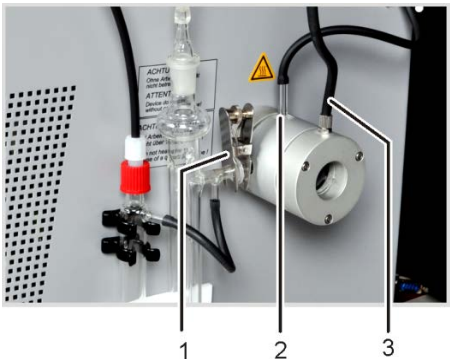
CAUTION

There is a risk of burning on the combustion furnace! Allow the device to cool down sufficiently before removing 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 with Ethanol before installing it in the combustion furnace.

1. Switch the basic device off at the main switch, pull the mains plug from the mains outlet and disconnect the gas supply.

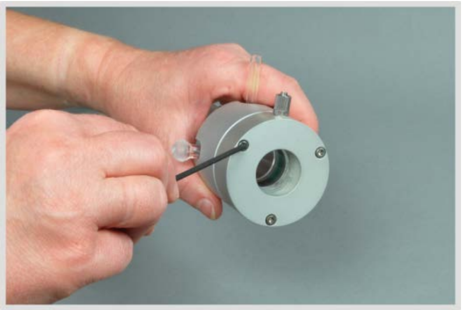


1	fork clamp
2	oxygen supply connection
3	argon connection

2. Pull the glass olive out of the sulfuric acid container. If using the safety container, pull the hose of the safety container off the bypass of the acid container.
3. Remove the fork clamp (1) and release the spherical joint between the gas outlet of the quartz tube and the sulfuric acid container.
4. Carefully remove the acid container and drain it.
5. Pull the gas hose off the gas inlet connection of the quartz tube (2).
   If using the flame sensor unscrew it (see also section "Connecting the flame sensor

(optional)" p. 61).

6. Undo the union nut at the argon connection of the gas airlock (3) and pull off the hose.
7. Grip the gas airlock and pull the quartz tube out of the combustion furnace.



8. Unscrew the hexagon socket screws and dismantle the gas airlock.
9. Using a hook, pull the quartz wool out of the tube.
10. Check the quartz tube for excessive crystallization, cracks and blown out places.

    Note:
    Only re-use intact combustion tubes.
11. Flush the tube and dry it.
12. Fill approx. 2.5 cm quartz wool at the entrance of the quartz tube and, using a hook, push it to the tube end.
13. Then assemble the gas airlock and install the combustion tube in the furnace. Restore all gas connections (see section "Preparing the multi EA 4000 for Cl measurements" p. 59).

8.3.3 Servicing the combination electrode

CAUTION

Potential destruction of the combination electrode from detergent or abrasive agent.

For cleaning, the combination electrode may only be flushed with Ethanol or distilled water. The use of aggressive detergents or abrasives is prohibited.

If the electrolyte dries on the combination electrode this may cause an irreversible reduction of the sensitivity or damage of the electrode. Therefore, make sure that the electrolyte never dries on the combination electrode:

- during a brief pause in operation (from one day to the next):
  store the electrode in the electrolyte solution.
- during decommissioning for several days:
  rinse the combination electrode with ultrapure water and ethanol. Leave the electrode to stir in ethanol overnight. Then wipe the combination electrode with cellulose and store it dry.

IMPORTANT

The combination electrode consists of ceramic material and is especially sensitive in the electrode smelting area.

Handle the combination electrode with great care! As a wearing part it is not covered by warranty!

Pulling it out of the cover of the measuring cell and connecting it to the measuring gas can cause major mechanical stress if handled incorrectly and with it the fracture at the electrical connection of the combination electrode.

Correct	Incorrect
 Grip the electrode from above and pull the electrode straight up out of the cover.	 Do not pull and wobble at the connection sleeve for the electrical connections on the side. The connections in the sleeve will break (not visible from outside)!

Correct	Incorrect
 Slide the measuring hose only approx. 1 cm onto the electrode. This achieves gas-tightness.	 If you slide the hose too far onto the electrode, it gets stuck on the electrode. The electrode can break when the hose is pulled out!

8.3.4 Servicing the measuring cells

Since the entry of warm reaction gases during the measurement results in the evaporation of electrolyte components, it is recommended to replace the electrolyte solution daily:

1. Remove the used-up electrolyte.
2. Rinse the measuring cell and the magnetic stirring rod with water or ethanol.
3. Next carefully wipe the measuring cell and the magnetic stirring rod with cellulose to remove any sliver chloride deposits present.
4. Re-fill the measuring cell with electrolyte (see section "Making the measuring cell available").

Observe the following notes:

- If the system is decommissioned for several days, clean the measuring cell and store it dry.
- Check the outer sleeve of the magnetic stirring rod regularly for cracks. Metal ions entering the electrolyte solution may interfere with the analysis.
- Avoid the ingress of liquid into the stirring/cooling block and the plug-in contact (danger of short circuit).

8.3.5 Replacing the adsorption agent

1. Switch off the basic device and the Cl module from the mains switch. Switch off the gas supply.
2. Pull the adsorption tube out of the holder.
3. Unscrew the screw caps from the glass tube.
4. Remove the cotton plugs and spill the adsorption agent.
5. Seal one side of the glass tube with cotton wool and fill the adsorption agent.
6. Seal the glass tube with cotton wool.
7. Screw the filled adsorption tube back on finger-tight.
8. Press the adsorption tube back into its holder.

Caution:
Do not twist or kink the hoses.

8.4 Operation with automatic TIC solids module

Maintenance intervals

Maintenance task	Maintenance interval
Remove chemical splashes from the housing	immediately
Clean the TIC reactor	daily, in case of visible contamination
Replace the desiccant in the drying tube	as required (blackening or clumping due to moisture absorption)
Replace the halide trap	if necessary, if half of the copper wool or the brass wool is discolored
Check the condensation coil for damage	after 3 months
Clean the condensation coil	as required, but no later than after 12 months
Check the condensate container for damage	after 3 months
Clean the condensate container	as required, but no later than after 12 months
Replace metering pump, pump hose	after 3 months
Replace condensate pump, pump hose	after 3 months

Replace the desiccant in the drying tube in the same manner as in the basic device multi EA 4000 (→ see section "Replacing the desiccant in the drying tube" p. 88).

Also replace the copper and brass wool in the halide trap analog to the replacement of the halide trap in the basic device in the multi EA 4000 (→ see section "Replacing the halide trap" p. 89).

8.4.1 Clean the TIC reactor of the automatic TIC solids module

WARNING

The phosphoric acid or hydrochloric acid used to purge the TIC can cause severe chemical burns!

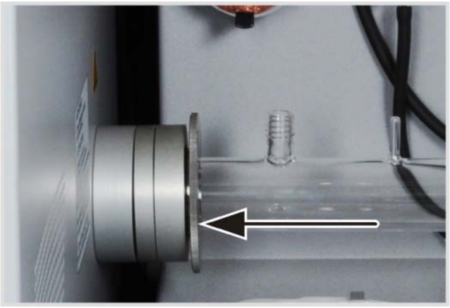
Always wear protective gloves and goggles when handling concentrated acid! Rinse spillages on the skin immediately.

1. Switch off the basic device and the TIC module from the mains switch. Switch off the gas supply.
2. Allow the device to cool down sufficiently (at least 2h).
3. Move the FPG 48 approx. 30 cm to the right to make space for removing the TIC reactor.

Caution: Support the reactor with one hand when removing the hoses from the connections. Pressure from one side might break the reactor. It is also possible to first pull the reactor out of the coupling and then remove the connections.

4. Unscrew the connection for the acid supply. Remove the ceramic cannula from the connection.
   Caution! Make sure that the acid does not drip from the hose onto the device.
5. Rinse the ceramic cannula with ultrapure water and wipe it clean with cellulose.

   Caution! The ceramic cannula is very fragile!
6. Pull off the argon and oxygen supply hoses.
7. Put the acid container and drip tray aside.



8. Whilst holding the connection rings rotate the knurled ring counterclockwise. This loosens the seal around the reactor.
9. Pull the reactor out of the seal of the connection rings.
10. Check the reactor for cracks and bursts.

    Note:
    Only use intact reactors.
11. Clean the reactors with a suitable detergent and brush or cellulose, if necessary.
12. Rinse the reactor with ultrapure water and dry it.
13. Slide the reactor back into the coupling and secure it by tightening the knurled ring.
14. Attach the acid and gas connections to the reactor and re-install the FPG 48 (see section "Connect the reactor and sampler FPG 48" p. 73 and "Retrofitting the sampler " p. 142).

8.4.2 Cleaning the condensation coil

1. Switch the basic device and the automatic TIC solids module off at the mains switch, pull the mains plug of the TIC solids module from the mains outlet and disconnect the gas supply.
2. Push the FPG 48 aside, remove the acid container and drip tray and remove the TIC reactor.
3. Rotate the TIC solids module until to gain access to the rear.

4. Pull the hoses off the condensation coil.
5. Carefully pull the condensation coil out of its holder.
6. Check the condensation coil for deposits and cracks.
7. If required, rinse the condensation coil with ultrapure water and leave to dry well.
8. Press the condensation coil back into the holder on the backplate of the TIC solids module.
9. Connect hose no. 72 (from the reactor) to the top connection of the condensation coil.
10. Connect hose no. 73 (to the reactor) to the bottom connection of the condensation coil.
11. Rotate the TIC solids module back into its original position.
12. Re-assemble the TIC reactor and the FPG 48 (see section "Connect the reactor and sampler FPG 48" p. 73 and "Connecting and using the sampler " p. 137).

8.4.3 Removing and replacing the pump hose

Acid pump

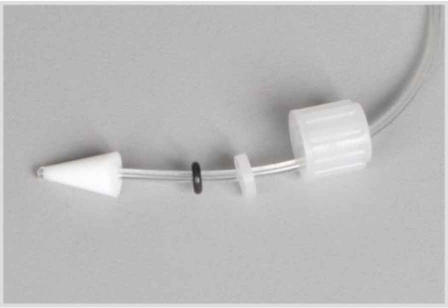
WARNING

The pump hose contains phosphoric acid or hydrochloric acid (dependent on the application). The acid is irritating to eyes, skin and mucous membranes!

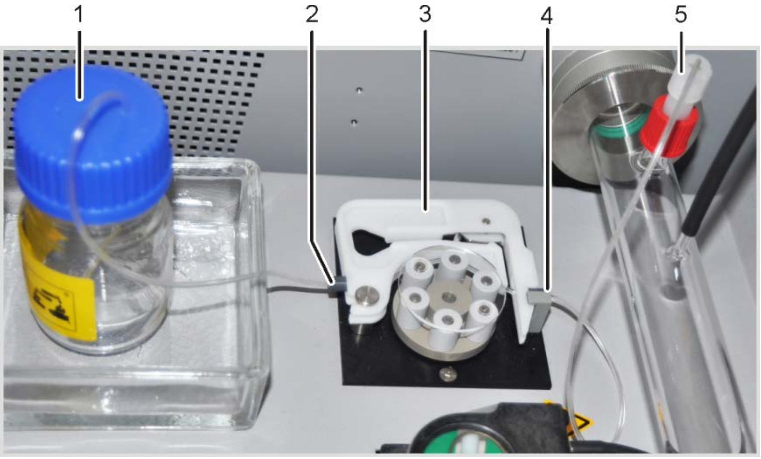
Always wear protective gloves and goggles when handling concentrated acid! Rinse spillages on the skin immediately.

Replace the pump hose at the cartridge pump as follows:

1. Pull the hose off the acid bottle. Put the acid bottle and drip tray aside.
2. Unscrew the pump hose from the reactor.
3. Open the hose cartridge and remove the pump hose.



4. Pull the sealing elements and the white union nut off the worn pump hose.
5. Slide the sealing elements in the sequence shown onto the new pump hose.



1	phosphoric acid bottle
2, 4	stopper at the pump hose
3	hose cartridge
5	hose connection at the TIC reactor

6. Clamp the pump hose between the two plastic stoppers into the hose cartridge.
7. Insert the hose cartridge into the pump and allow it to engage.
8. Screw the pump hose to the reactor.
9. Place the acid container in the drip tray onto the TIC module. Slide the other end of the pump hose into an opening of the acid container.

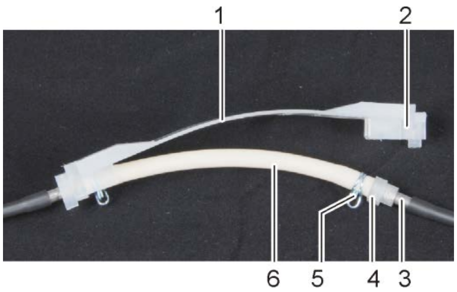
Condensate pump

Check the pump hose every three months for leaks. Replace the pump hose annually.

1. Push the bracket on the condensate pump to the left.
2. Pull the two hoses no. 77 and no. 78 off the connections.
3. Remove the conveyor belt with the pump hose from the pump body.
4. 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.
5. Wipe the pump body and roller carrier with ultrapure water.
6. Check the pump body and roller carrier for wear.

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



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

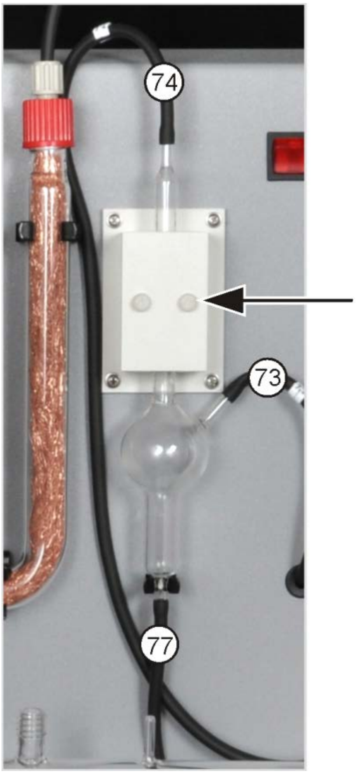
7. Push the faultless or new pump hose back into the conveyor belt.

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

8. Position the conveyor belt around the pump body.
9. Press the conveyor belt upwards with one hand and rotate the bracket with the other hand to the right until it engages.
10. Push hoses 77 and 78 back onto their adapters.

8.4.4 Cleaning the condensate container

Clean the condensate container as required:



1. Undo the 2 knurled head screws (arrow) at the cover of the cooling block and remove the cover.
2. Pull the hoses off the connections of the condensate container.
3. Check the condensate container for deposits and cracks.
4. If required, rinse the condensate container with ultrapure water.
5. Attach the hoses as shown in the adjacent figure:
    - Hose no. 73 (from the bottom end of the condensation coil on the rear) at the side entrance of the condensate container
    - Hose no. 77 (to the condensate pump) at the bottom outlet
    - Hose no. 74 (to the drying container) at the top outlet

    Slide the hoses approx. 1 cm wide onto the glass connections of the condensate container.

8.5 Operation with manual TIC solids module

Maintenance intervals

Maintenance task	Maintenance interval
Remove chemical splashes from the housing	immediately
Clean TIC reactor (Erlenmeyer flask)	between samples
Replace the desiccant in the drying tube	as required (blackening or clumping due to moisture absorption)
Replace the halide trap	if half of the copper wool or the brass wool is discolored
Replace the water traps	as required, but no later than after 3 months

Replace the desiccant in the drying tube in the same manner as in the basic device multi EA 4000 (→ see section "Replacing the desiccant in the drying tube" p. 88).

Also replace the copper and brass wool in the halide trap analog to the replacement of the halide trap in the basic device in the multi EA 4000 (→ see section "Replacing the halide trap" p. 89).

8.5.1 Clean the TIC reactor at the manual TIC solids module

After each analysis rinse the TIC reactor (Erlenmeyer flask) with ultrapure water. Then dry it thoroughly (see also "Performing the TIC measurement" p. 80).

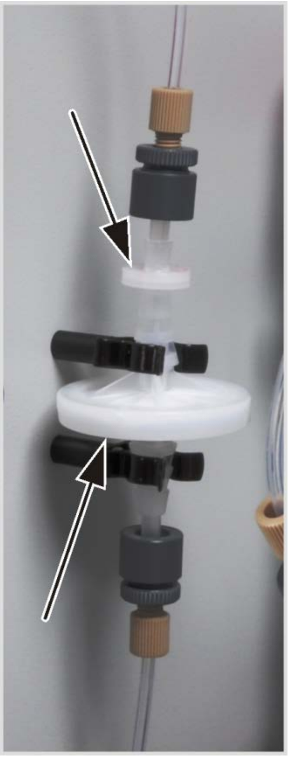
8.6 Replacing the water traps

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. Pull the water traps out of the clamps. 2. Pull the hose connections out of the water traps. 3. 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) 4. Attach the large water trap to hose no. 8. 5. Connect the small water trap to hose no. 9. 6. Press the water traps into the clamp on the equipment backplate.

9 Fault removal

9.1 General information

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 storage location for the log files can be defined via the menu EXTRAS / CONFIGURATION tab FAULT ANALYSIS.

The default storage locations for the multiWin® specific log files multiWin_Exception.log and multiWin_trace.log are

- in Windows XP:
  C:\Documents and settings\All Users\Application data\ajIDC\multiWin\
- in Windows Vista/Windows 7:
  C:\ProgramData\ajIDC\multiWin\

Default storage locations for the device-specific log files multiWin_ERROR.log (recoding is always enabled by default), multiWin_Comm.log and multiWin_Flow.log are

- in Windows XP:
  C:\Documents and settings\All Users\Application data\ajIDC\multiWin\..\device number
- in Windows Vista:
  C:\ProgramData\ajIDC\multiWin\...\device number

IMPORTANT

If faults cannot be remedied by the customer, the Analytik Jena AG service department must always be informed. This also applies for the repeated occurrence of individual faults.
For the fault diagnosis the respective files must be sent by email to the Analytik Jena AG service department.

9.2 Error messages in multiWin

	Error message	
1	**No reply from firmware!**	
	Cause	**Remedy**
	control module and basic device not switched on	– switch on control module and basic device
	control module not connected to PC	– check connection between control module and PC
	incorrect interface selected on PC	– check connected interface on PC – if necessary select different interface in multiWin via the menu item EXTRAS / INTERFACE – initialize

2	**Serial port not present!**	
	Cause	**Remedy**
	communication problems	– disconnect USB connection between control module and PC and reconnect after approx. 10 s – initialize
3	**Serial port not reachable!**	
	Cause	**Remedy**
	communication problems	– disconnect USB connection between control module and PC and reconnect after approx. 10 s – initialize
4	**Unknown connection type!**	
	Cause	**Remedy**
	communication problems	– disconnect USB connection between control module and PC and reconnect after approx. 10 s – initialize
5	**Error when opening the port!**	
	Cause	**Remedy**
	communication problems	– disconnect USB connection between control module and PC and reconnect after approx. 10 s – initialize
7	**Operating system error: Unauthorized access!**	
	Cause	**Remedy**
	undefined termination of multiWin	– exit multiWin – disconnect USB cable and reconnect after approx. 10 s – restart operating system (PC) – restart firmware (basic module) – restart multiWin
11	**no reply to signal**	
	Cause	**Remedy**
	firmware crashed	– exit multiWin – switching off the analyzer – disconnect USB cable between PC and control module reconnect after approx. 10 s – restart operating system (PC) – switch on analyzer – start multiWin
12	**Signal echo received, check port selection**	
	Cause	**Remedy**
	incorrect interface selected	– check interface selection

14	**Data transfer interrupted**	
	Cause	**Remedy**
	no data transfer for 10 s	– initialize
17	**Incorrect interface protocol ID**	
	Cause	**Remedy**
	error following update (program versions firmware – multiWin® do not match)	– update required
20	**Timeout: Intend**	
	Cause	**Remedy**
	timeout during initialization	– initialize
21	**Timeout: Status Busy**	
	Cause	**Remedy**
	timeout during operation (device not ready for measurement)	– acknowledge message – initialize
22	**Timeout: End**	
	Cause	**Remedy**
	timeout when exiting multiWin	– acknowledge message – initialize
23	**Timeout: Stop End**	
	Cause	**Remedy**
	timeout during measurement cancellation	– acknowledge message – initialize
24	**Timeout: Measurement**	
	Cause	**Remedy**
	timeout during measurement maximum integration time exceeded	– check analyzer – check method configuration
25	**Timeout: Analyzer command processing**	
	Cause	**Remedy**
	timeout during command processing	– acknowledge message – initialize
30	**Chloride needs adding**	
	Cause	**Remedy**
	chloride needs adding	– open menu item SYSTEM / COMPONENT TEST
50	**Firmware reset**	
	Cause	**Remedy**
	internal computer (firmware) has restarted.	– acknowledge message – initialize
52	**Firmware standby**	
	Cause	**Remedy**
	firmware was run in standby	– acknowledge message – initialize

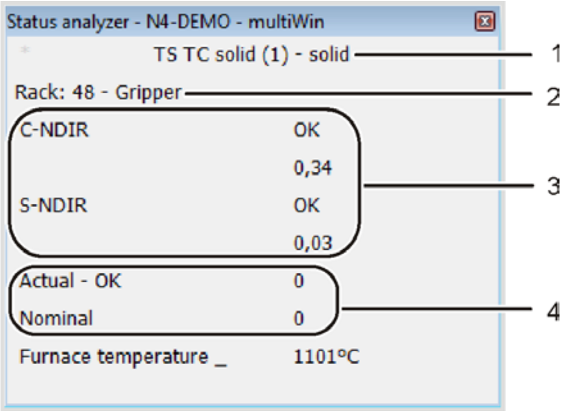
61 **62** **64** **65** **66** **67**	**Command from the PC not complete** **Command from the PC without STX** **Command from the PC CRC error** **Command from the PC invalid command** **Command from the PC invalid MEAS command** **Command MTXT from PC missing**	
	Cause	**Remedy**
	communication error	– acknowledge message – initialize
100	**C sensor: no connection**	
	Cause	**Remedy**
	communication faulty after sensor was detected during initialization	– acknowledge message – initialize
101	**C sensor: CRC error**	
	Cause	**Remedy**
	communication faulty after sensor was detected during initialization	– acknowledge message – initialize
104 **107**	**C sensor: Analog values out of range** **C sensor: Analog values out of range (coarse)**	
	Cause	**Remedy**
	the analog values of the sensor are outside the operating range	– check quality of carrier gas – initialize – check analog values in the component test (via menu item SYSTEM / COMPONENT TESTacy-NDIR
106	**C sensor: incorrect response to command**	
	Cause	**Remedy**
	communication interrupted	– switch QC module off and on again – initialize
110 **120** **130**	**N sensor: no connection** **S sensor: no connection** **Cl sensor: no connection**	
	Cause	**Remedy**
	communication faulty after sensor was detected during initialization	– acknowledge message – initialize
120	**S sensor: no connection**	
	Cause	**Remedy**
	communication faulty after sensor was detected during initialization	– acknowledge message – initialize
124	**S sensor: incorrect status**	
	Cause	**Remedy**
	change to smoothing factor / LSCM switch-on failed	– repeat action
128	**S sensor: inactive**	
	Cause	**Remedy**
	sensor type transmission failed	– repeat action

130	**Cl sensor: no connection**	
	Cause	**Remedy**
	communication faulty after sensor was detected during initialization	– acknowledge message – initialize
131	**Cl sensor: incorrect command structure**	
	Cause	**Remedy**
	communication to chlorine module faulty	– acknowledge message – switch chlorine module off/on – initialize
133	**Cl sensor: incorrect cell**	
	Cause	**Remedy**
	no initialization after cell change	– initialize
134	**Cl sensor: incorrect status**	
	Cause	**Remedy**
	communication interrupted	– acknowledge message – initialize – check status of titration cell via menu item SYSTEM / COMPONENT TESTtabCHLORINE
135	**Cl sensor: incorrect version**	
	Cause	**Remedy**
	transmission fault	– acknowledge message – initialize – check status of titration cell via menu item SYSTEM / COMPONENT TEST / CHLORINE
137	**Cl sensor: check measuring cell**	
	Cause	**Remedy**
	end point routine unsuccessful 3 times	– check electrolyte filling – check electrodes
138	**Cl sensor: inactive**	
	Cause	**Remedy**
	sensor type transmission failed	– repeat action
139 **140**	**Cl sensor: STA error** **Cl sensor: STA error**	
	Cause	**Remedy**
	error during update of the Cl sensor	– repeat update
207	**Flow error**	
	Cause	**Remedy**
	flow state not OK.	– check for leaks – check gas supply
208	**O_2 pressure missing**	
	Cause	**Remedy**
	O_2 gas flow insufficient	– check O_2 connection – check pressure

209	**Ar pressure missing**	
	Cause	**Remedy**
	argon flow insufficient	– check argon connection – check pressure
220	**Sampler: no connection**	
	Cause	**Remedy**
	communication faulty after sampler was detected during initialization	– acknowledge message – initialize
226	**Sampler: runtime exceeded**	
	Cause	**Remedy**
	completion message of sampler movement takes too long (sampler faulty)	– record log files – inform Service
290	**Scales: no connection**	
	Cause	**Remedy**
	no communication with the scales	– check connection – check configuration of scales
291	**Scales: incorrect weight unit**	
	Cause	**Remedy**
	currently only one unit [g] is permissible	– set the unit gram [g] in the menu of the scales
300	**Temperature controller: no connection**	
	Cause	**Remedy**
	communication interrupted	– acknowledge message – initialize
0301	**Thermocouple break**	
	Cause	**Remedy**
	thermocoupler HT 1500 faulty	– contact Service
303 **304**	**Temperature controller: EPROM error** **Temperature controller: Communication error**	
	Cause	**Remedy**
	communication fault with the basic device	– switch basic device off/on – initialize
306	**external furnace not present**	
	Cause	**Remedy**
	basic device not detected as hardware	– check connection between basic device and control module

9.3 Indications in the window DEVICE STATE

In the window STATUS ANALYZER Information on the device status or individual modules is displayed.



Status analyzer - N4-DEMO - multiWin

TS TC solid (1) - solid

Rack: 48 - Gripper

C-NDIR OK

0,34

S-NDIR OK

0,03

Actual - OK 0

Nominal 0

Furnace temperature _ 1101°C

1	method
2	analysis module / sampler
3	detection/sensor module
4	flow indication (not for Cl measurements)
5	temperature indication

Fig. 33 **Window STATUS ANALYZER**

The displays in the STATUS ANALYZER window are indicated in color. The colors have the following meanings:

Color	Description
Black	Status of the corresponding component is ok, device is ready for measurement
Gray	Detector is inactive
Green	Detector is ok, device is ready for measurement (OK) or Detector is busy, a measurement can only be started once the routine is complete (detector-specific)
Red	Component is not ready for measurement Warm-up time not yet complete ⇒ wait until warm-up time is complete Error ⇒ Error search, find information on corresponding components in multiWin via the menu item SYSTEM / COMPONENT TEST

In the top line of the window STATUS ANALYZER the name of the active method and the sample state (solid) are displayed.

During initialization it is detected whether the sampler FPG 48 is connected and switched on. In this case the display "Rack 48" appears. A connected automatic TIC solids module is also detected and displayed as "TIC (automatic)".

All the detector modules that are identified during initialization are displayed in the Detection/Sensor Module area. The following displays are possible:

Display	Description
C-NDIR	NDIR detector for carbon
S-NDIR	NDIR detector for sulfur
Cl-AMP smallCell	Chlorine module with "sensitive" cell
Cl-AMP largeCell	Chlorine module with "high concentration" cell

The respective status of the detector modules is indicated by color (see above). The following status displays are possible:

Device ready for measurement	
Display	**Description**
OK (green, black)	Detector is ready for measurement
Device not ready for measurement - general	
Display	**Description**
no detector displayed (display empty)	No detector identified: – Activate detector – initialize
Communication error (rot)	Communication interrupted: – Deactivate/activate the device – initialize
No connection (red)	Connection interrupted: – Check connection cable – Deactivate/activate the device – initialize
Device not ready for measurement - C-NDIR / S-NDIR	
Display (rot)	**Description**
Warning, analog values	Analog values outside range: – Read the values via menu item SYSTEM / COMPONENT TEST on the tab C-NDIR, S-NDIR – see also error descriptions for the C/S module S. 119
Warm-up time	Detector is not yet ready for operation: Wait until the warm-up time is complete after activation (approx. 30 min)
Device not ready for measurement - Cl-AMP	
Display (rot)	**Description**
Inactive	No cell identified: Use cell
Device not ready for measurement - Cl-AMP	
Display (green)	**Description**
Interval titration	Interval titration running: – Possible to start measurement
End point routine	Limit point routine running: – Wait until the limit point routine has finished

In the area of the flow indication the current intake flow (ACTUAL) and the target flow to be reached (TARGET) are displayed. The target flow is 100 l/h. The intake flow may differ ± 10 l/h from the target flow. Adjust the intake flow at the rotameter "pump" at the front panel of the basic device.

The temperature display shows the current furnace temperature. If the temperature display is red, the current furnace temperature does not match the temperature set in the method. In this case wait until the target temperature has been reached or check the temperature configuration in the method.

9.4 Equipment fault

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

If the suggested solutions are not successful, inform Service.

9.4.1 Basic device

Error	
Furnace does not heat	
Cause	**Solution**
– electronics malfunction – heating rod broken	– check the electrical plug-in connection – check the internal fuse – inform Service
Fans on the rear of the device are not running	
Cause	**Solution**
– fuse faulty – electronics faulty	– switch analyzer off from the main switch – inform Service
No intake flow at flow meter "pump"	
Cause	**Solution**
– pump not switched on	– check temperature difference – Wait for the temperature difference between target and actual temperature to approximate 50 °C. If the pump does not switch on with a temperature difference between target and actual temperature of less than 50 °C, inform Service.
– hoses disconnected	– check hose system for kinked or squeezed hoses

9.4.2 C/S module

multiWin: flow error **measuring gas flow ACTUAL< 90 l/h or >110 l/h**	
Cause	**Solution**
– no or insufficient intake flow	– With the intake pump switched on correct the intake flow at the flow meter "pump" in the basic module. – check hose system for kinked or squeezed hoses
– desiccant too wet and baked together	– replace desiccant

multiWin: flow error
measuring gas flow ACTUAL = 0

Cause	Solution
– no intake flow – pump not switched on	– check temperature difference – Wait for the temperature difference between target and actual temperature to approximate 50 K. If the pump does not switch on with a temperature difference between target and actual temperature of less than 50 K, inform Service.

Analog values of NDIR optics fluctuate

Cause	Solution
– leaks – configuration or fault of the NDIR detector – detector stabilization during warm-up phase of the analyzer not yet complete	– wait for warm-up phase of 15 min. – If the analog values are not constant after 1 h (whilst oxygen is present), a leak may be present. inform Service – Replace halide trap, if discolored, to protected the detector.

multiWin: Optical error, analog value out of range
Values of the NDIR detector are shown in yellow in the status window

Cause	Solution
– The ADU values of the NDIR detector differ from the target value and are outside of the tolerance ranges Note: Measurements are still possible.	– check the oxygen supply of the gas supply – check the oxygen supply at the flow meter, open the valve – check the oxygen flow through the detector (hose obstruction) – inform Service

multiWin: no connection to the optics (NDIR)
multiWin: Optics status error

Cause	Solution
– communication error – NDIR detector faulty	– reinitialize the device. – inform Service

Control indicator light is not illuminated

Cause	Solution
– internal program has not been started	– Restart the internal computer with the mains switch of the module.

IMPORTANT

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

9.4.3 Cl module

IMPORTANT

To prevent errors always note:

- Connect measuring cell with stirrer rod (filled with electrolyte) and electrode before switching on the analyzer.
- Start the multiWin software approx. 3 min after switching on the analyzer.

multiWin: no connection to the Cl sensor	
Cause	**Solution**
– No communication between internal computer and coulometer. – Cl module not switched on	– Observe switching-on sequence: 1. switch on basic unit 2. switch on Cl module 3. Wait approx. 3 min until the coulometer communicates with the internal computer. 4. start the multiWin software
– connection cable not connected or faulty	– check cable connection
multiWin: wrong command structure Cl sensor	
Cause	**Solution**
– fault during data transmission coulometer – internal computer	– re-initialize analyzer – inform Service
multiWin: indication error	
Cause	**Solution**
– electrode connector not connected – electrode faulty	– connect electrode and re-initialize device – replace faulty electrode – inform Service

9.5 Device communication errors

Software does not respond.	
Cause	**Solution**
– Computer crash	– Switch off the analyzer completely, incl. basic device, printer, sampler and external PC, and restart the system after few seconds.
multiWin: communication error– analyzer	
Cause	**Solution**
– module with internal computer not switched on – multiWin started too early – module with internal computer not connected to PC – Incorrect COM interface connected at the external computer or selected in multiWin	– switch on the module – check connection between module and internal computer – only start multiWin after the LED indicator LOCK IN illuminates – check the interface connected at the external computer and configured in multiWin (menu command **Configuration / Interface / Interface selection**)
during the running operation of the device together with error **multiWin: Restart computer in the analyzer**	
Cause	**Solution**
– internal computer reset	– If front LED illuminates, initialize analyzer – If error occurs repeatedly, monitor timing (status line) and inform Service.
multiWin: Communication error - incorrect command set between PC and device	
– the internal and external program versions do not match.	– repeat update
multiWin: no connection to the temperature controller	
Cause	**Solution**
– basic device not switched on – Interface cable between module and internal computer and basic device not connected or faulty. – Temperature controller faulty.	– switch on basic device and re-initialize multiWin 1 min. later – check connection of the interface cable
No connection to the autosampler	
Cause	**Solution**
– no communication between basic device and sampler – sample not switched on – connection cable not connected or faulty	– check connection cable – switch on sampler and initialize analyzer

9.6 Analytical problems

9.6.1 C/S detection

Recovery generally too low	
Cause	**Solution**
– Drying agent too moist – Filter and measuring gas hoses sooted up	– Check and replace the desiccant – Check and replace the filter – Check dust trap, if heavily contaminated (particle deposits), clean dust trap and replace measuring gas hose up to the filter
Too low results in TS	
Cause	**Solution**
– Halogen trap still in measuring gas flow	– Replace halogen trap with an empty glass tube
– Digestion temperature too low (especially for sulfate bonding form)	– Increase furnace temperature in the method, if S measuring graph look very irregular or has a long trailing and does not reduce to the base line. – If necessary, add additives (e. g. iron phosphate, tungsten oxide, vanadium pentoxide, tin…).
– Contamination of the hoses carrying the measuring gas up to the optics	– Remove hoses up to the NDIR optics, clean, dry and re-install them. **Note**: We recommend to have the cleaning of the hoses carried out by Service.
Excess results during TC	
Cause	**Solution**
– Combustion tube has a crack or is broken	– Ambient air can enter through the cracks into the system and may cause significant excess results in TC. ⇒ Replace combustion tube
Base line drift	
Cause	**Solution**
– Detector not stable	– The detector must stabilize during the warm-up phase of the device. Therefore, it may happen at the start of measuring that the base line still drifts a little (temperature dependence). The peak area changed due to the drift will be corrected automatically, thus the measuring results are never affected. – If the drift does not stop, inform Service.

9.6.2 Cl detection

IMPORTANT

To prevent errors always note:

- Connect measuring cell with stirrer rod (filled with electrolyte) and electrode before switching on the basic device and Cl module.
- Start the multiWin program approx. 3 min after switching on the analyzer.

Recovery too low (splitting factor > 1.4)	
Cause	**Solution**
– sulfuric acid too diluted	– check and replace sulfuric acid
– components or measuring gas hoses sooted up	– Check combustion tube for contamination. Clean the combustion tube by burning off the deposits with a Bunsen burner or in the muffle furnace at 1000 °C. Replace heavily contaminated tubes. Replace the quartz wool in the combustion tube – Check sulfuric acid container for soot. If particles have entered as far as the sulfuric acid container, clean the container and connection at the furnace outlet and refill the container.
Recovery too high (splitting factor < 0.6)	
Cause	**Solution**
– Sample deposits in the combustion tube (e. g. salts)	– Replace or clean combustion tube, e.g. by "soaking" the combustion tube overnight in 10% HNO_3, then rinse thoroughly with distilled water and dry.
Shutdown threshold below 1500	
Cause	**Solution**
– Combination electrode faulty or too old	– Use new electrode for chlorine detection.

9.6.3 TIC detection (automatic TIC solids module)

Lower results	
Cause	**Solution**
– Weighted sample chosen too high	– Weighted sample should not exceed 50 mg, otherwise the volume of metered acid (400 µl) is not enough to completely replace the carbonate or the acid volume cannot fully wet the sample.
– Sample not distributed evenly over the entire length of the boat	– Sample must be distributed evenly to allow the sample to be completely wetted with acid.
– Acid strength too weak (long trailing of measuring graph, graph does not return to base line)	– If the sample contains larger amounts of very stable carbonates (e. g. $FeCO_3$), the breakdown speed with 40% H_3PO_4 is too slow. In this case use stronger acid (e.g. 25% HCl). **Caution** HCl may not be used permanently.
Scattering measurements	
Cause	**Solution**
– Sample not distributed evenly over the entire length of the boat	– Sample must be distributed evenly to allow the sample to be completely wetted with acid.
– Sample matrix is too hydrophobic	– If it is difficult to wet the sample with acid, it helps to cover the sample with annealed quartz sand to improve the effect of the acid. – The wetting properties of hydrochloric acid are sometimes better than those of phosphoric acid. **Caution** HCl may not be used permanently.

9.6.4 TIC detection (manual TIC solids module)

Lower results	
Cause	**Solution**
– Acid strength too weak (long trailing of measuring graph, graph does not return to base line with max integration time of 600 s)	– If the sample contains larger amounts of very stable carbonates (e. g. $FeCO_3$), the breakdown speed with 40% H_3PO_4 is too slow. In this case use stronger acid (e.g. 25% HCl). **Caution** HCl may not be used permanently.

9.6.5 Measurements in pyrolysis mode (EC)

<table>
<tr><th colspan="2">Results of elementary carbon too low (after pyrolysis)</th></tr>
<tr><th>Cause</th><th>Solution</th></tr>
<tr><td>– Waiting time to display the oxygen with argon in the combustion tube chosen too short</td><td>– Waiting time after changing from oxygen to argon supply should be at least 60 s.</td></tr>
<tr><th colspan="2">Results of elementary carbon too high (after pyrolysis)</th></tr>
<tr><th>Cause</th><th>Solution</th></tr>
<tr><td>– Pyrolysis time selected too short</td><td>– The pyrolysis time should be at least 3 min. to ensure that all organic compounds have been dissolved and purged completely.</td></tr>
<tr><td>– Pyrolysis temperature chosen too high</td><td>– At temperatures above 1000 °C soot is increasingly formed from organic compounds (plastics) through "coking", which is also detected in stage 2 as elementary carbon. The optimum furnace temperature for pyrolysis measurements is 850 °C.</td></tr>
</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 burns at the hot combustion furnace. Only remove the combustion tube 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!

ATTENTION! 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!

Prepare the EA 4000 and its modules for transport as follows:

Basic device multi EA 4000

1. Switch off the analyzer from the main switch and allow the equipment to cool down.
2. Disconnect the gas supply and unplug the mains plug from the mains outlet.
3. Undo all connections on the back of the analyzer.
4. Open the doors of the analyzer. Empty the drying tube. Reinsert the drying tube.
5. Unscrew the connections at the halogen trap and press the halogen trap out of the clamps.
6. Pull the combustion tube out of the furnace.
7. Carefully package the accessories, in particular protect glass components against breakage.

C/S module

1. Switch the C/S module off from the main switch.
2. Disconnect the power cable from the mains socket.
3. Undo all connections on the back of the module.

Cl module

1. Switch the Cl module off from the main switch.

Remove the measuring cell from the device and drain it (see section "

2. Servicing the measuring cells" p. 94.
3. Rinse and dry the measuring cell and the combination electrode.
4. Unscrew the adsorption tube and drain it.
5. Remove the halide trap.
6. Undo all connections on the back of the module.
7. Remove the sulfuric acid container and safety container from the basic device multi EA 4000 ab. Remove the sulfuric acid container (see section "Replacing the sulfuric acid/cleaning the sulfuric acid container" p. 90).
8. Pull the measuring gas hose (transparent) and the bypass hose (black) off the duct in the multi EA 4000.

Automatic TIC solids module

1. Switch the TIC solids module off from the mains switch at the front panel.
2. Undo all connections on the back of the module.
3. Disconnect the gas supply and unplug the mains plug from the mains outlet.

10.1.2 Transport notes

Observe the safety instructions in section "Safety instructions, transport and installation" p.13. 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 Moving the analyzer in the laboratory

CAUTION

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

Observe the following when moving the analyzer within the laboratory:

- Insufficiently secured components pose a risk of injury! Before moving the analyzer remove all loose components, in particular the reagent bottle with phosphoric acid and the sulfuric acid container.
- 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. 49.

10.2 Storage

CAUTION

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

The analyzer must only be stored in air-conditioned 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 Installing the multi EA 4000 C/S

Connections

For the combination of multi EA 4000 – and C/S module the following connections must have been made:

multi EA 4000 connections		Target
"valve"	➩	C/S module "valve"
"analyte"	➩	C/S module "analyte"
"O_2"	➩	Oxygen gas connection
Mains cable	➩	Mains connection
"COM"	➩	C/S module "COM"
"pump"	➩	C/S module "pump"
"Ar"	➩	Gas connection for argon (only required for Cl measurement and detection of carbon after pyrolysis)
"out"	➩	open output
additional C/S module connections		**Target**
"PC"	➩	USB connection for PC connection
"scale"	➩	external scales (optional)
"sampler"	➩	connection of the sampler FPG 48
socket for mains connector	➩	Mains connection

For the optional carbon detection after pyrolysis additional attach an exhaust pipe in the immediate vicinity of the furnace entrance.

Hose diagram

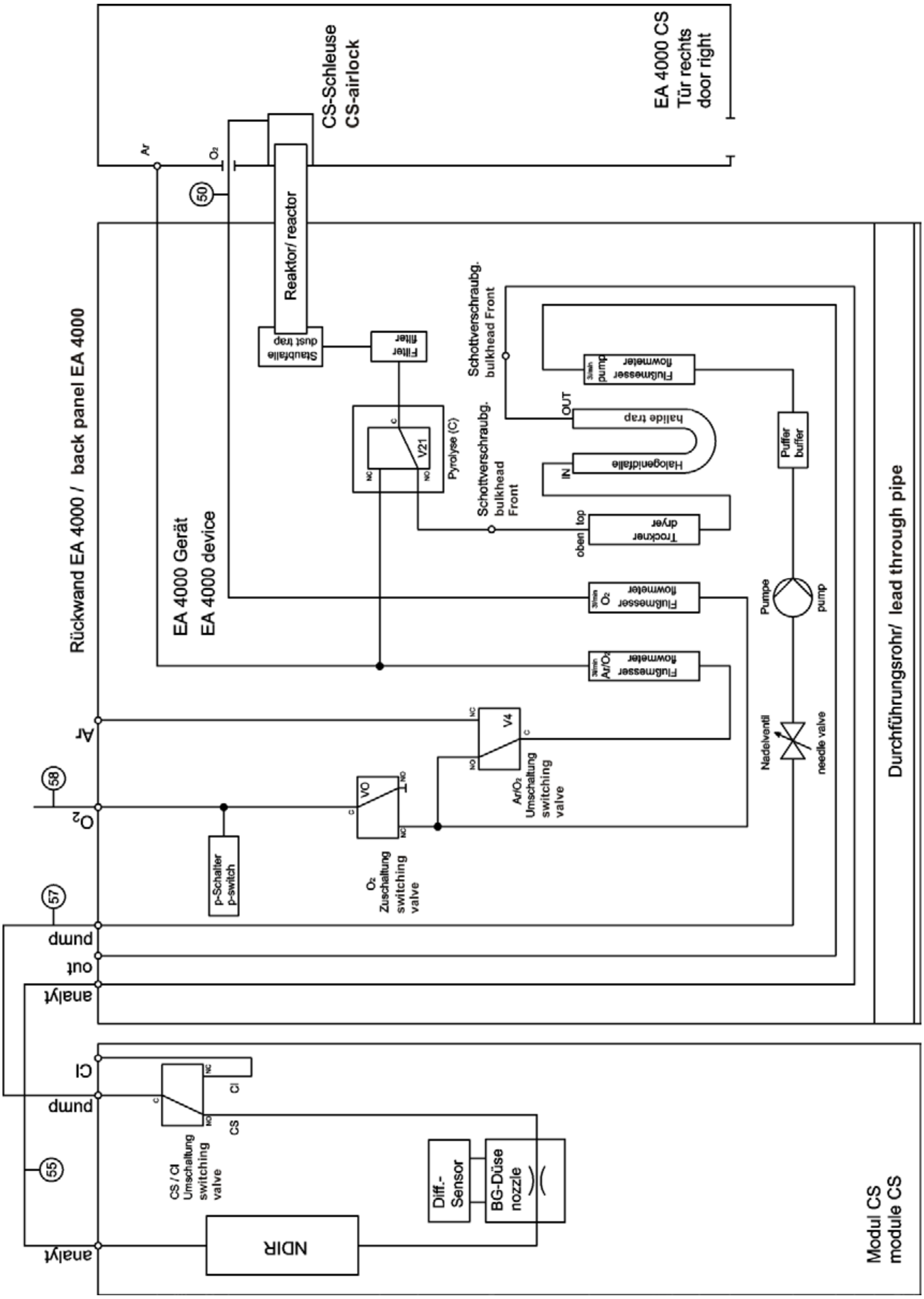


Fig. 34 Hose diagram for multi EA 4000 C/S

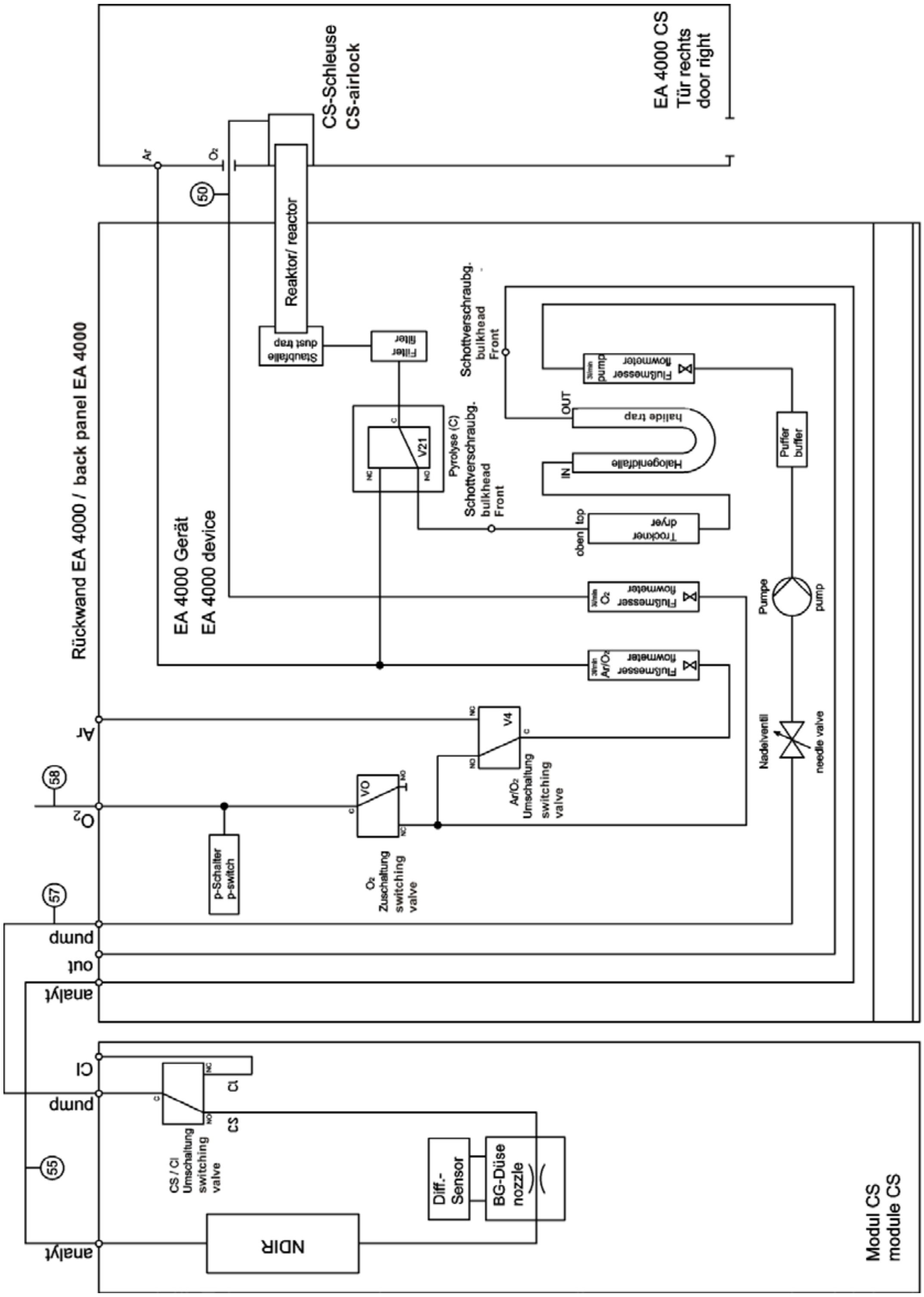


Fig. 35 Hose diagram multi EA C/S for measurement in pyrolysis mode

10.3.2 Installing the multi EA 4000 Cl

Connections

multi EA 4000 connections		**Target**
"valve"	➪	Cl module "valve"
"analyte"	➪	open output
"O_2"	➪	Oxygen gas connection
"Ar"	➪	Argon gas connection
Mains cable	➪	Mains connection
"COM"	➪	Cl module "COM"
"pump"	➪	Cl module "pump"
"out"	➪	open output
additional connections of the Cl module		**Target**
socket for mains connector	➪	Mains connection
"sampler"	➪	connection of the sampler FPG 48
"PC"	➪	USB connection for PC connection
"scale"	➪	external scales (optional)

Hose diagram

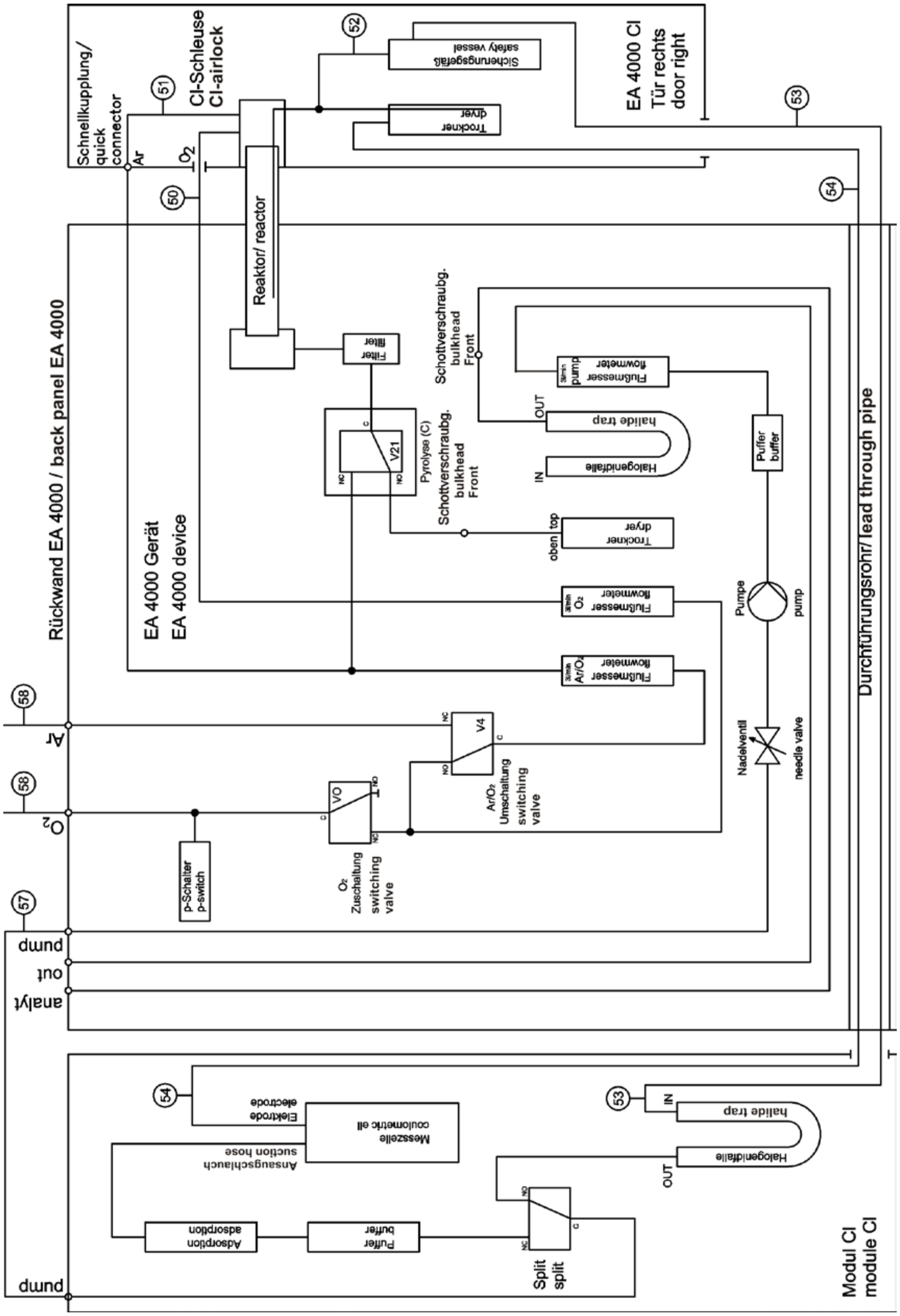


Fig. 36 **Hose diagram multi EA CI**

10.3.3 Installing the multi EA 4000 C/S Cl

Connections

For the combination of multi EA 4000 – and C/S module - Cl module the following connections must have been made:

multi EA 4000 connections		**Target**
"valve"	➪	C/S module "valve"
"analyte"	➪	C/S module "analyte"
"O_2"	➪	Oxygen gas connection
Mains cable	➪	Mains connection
"COM"	➪	C/S module "COM"
"pump"	➪	C/S module "pump"
"Ar"	➪	Gas connection for argon (only required for Cl measurement and pyrolysis)
"out"	➪	open output
additional C/S module connections		**Target**
"PC"	➪	USB connection for PC connection
"Cl-Coul"	➪	Cl module / "Cl-Coul"
"scale"	➪	external scales (optional)
"sampler"	➪	connection of the sampler FPG 48
socket for mains connector	➪	Mains connection
additional connections of the Cl module		**Target**
"Cl"	➪	C/S module "Cl"
socket for mains connector	➪	Mains connection

Hose diagrams

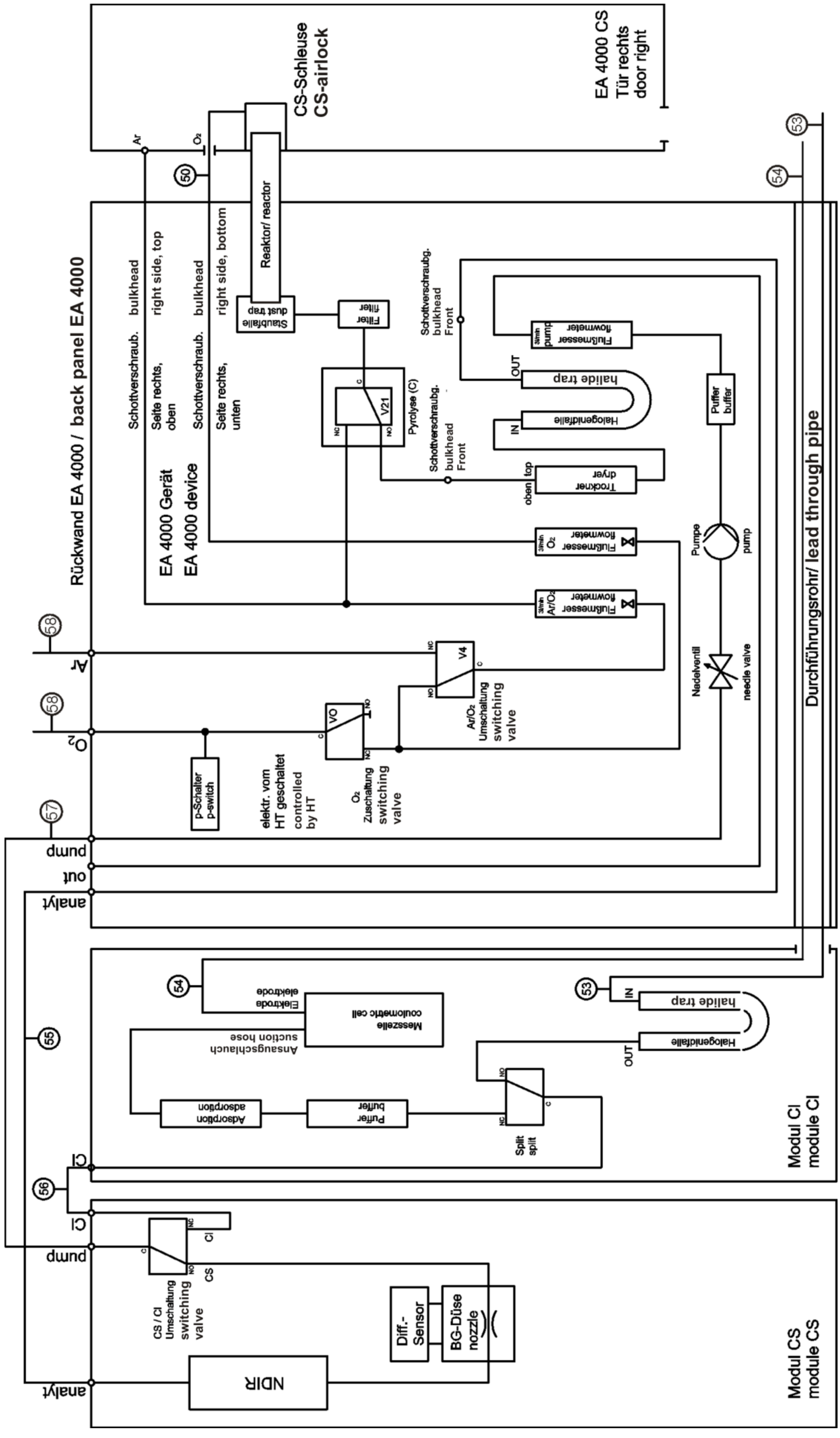


Fig. 37 Hose diagram multi EA C/S Cl for C/S measurements with ceramic tube and oxygen airlock

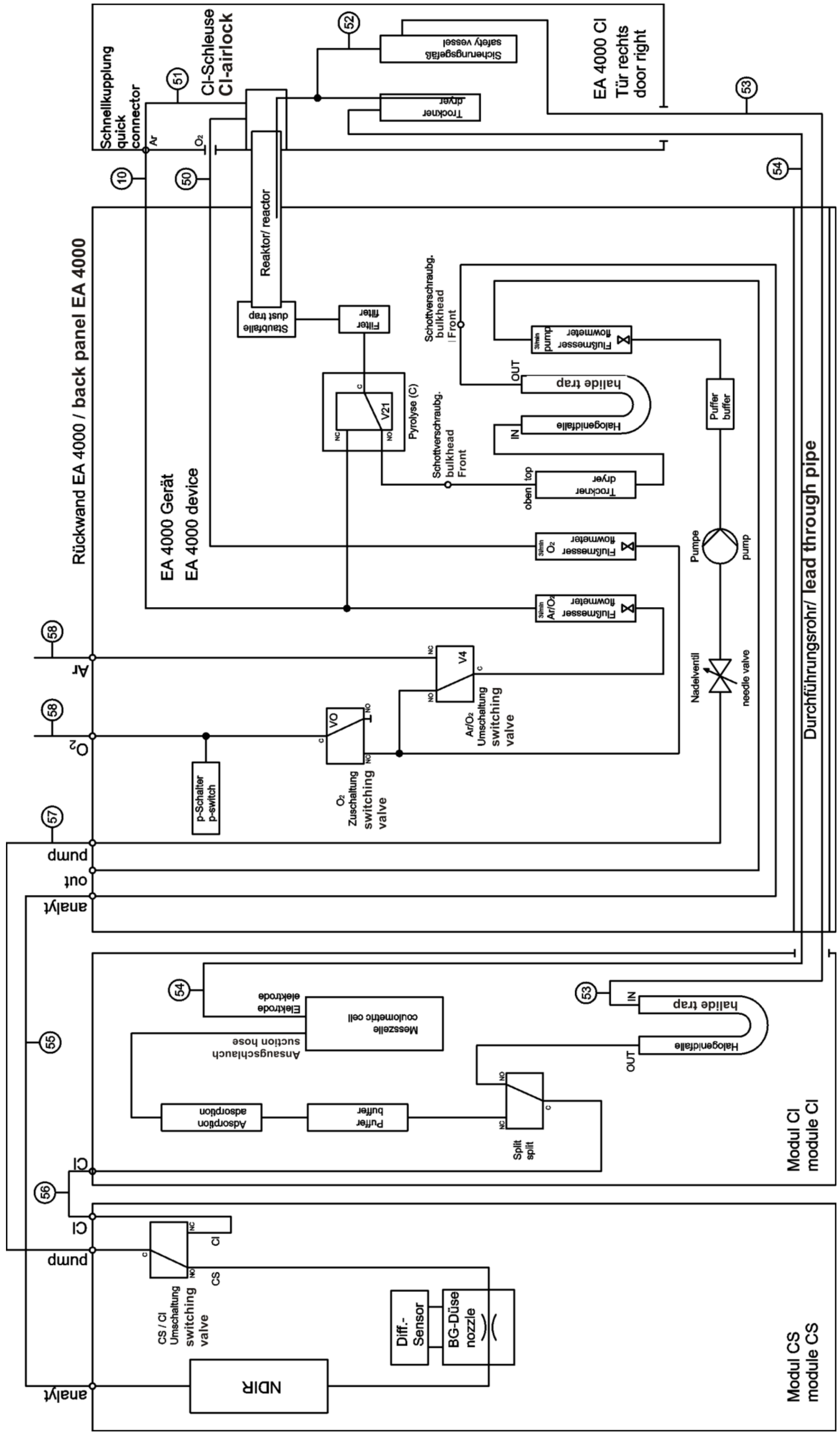


Fig. 38 Hose diagram multi EA C/S Cl for Cl measurements with quartz tube and argon connection

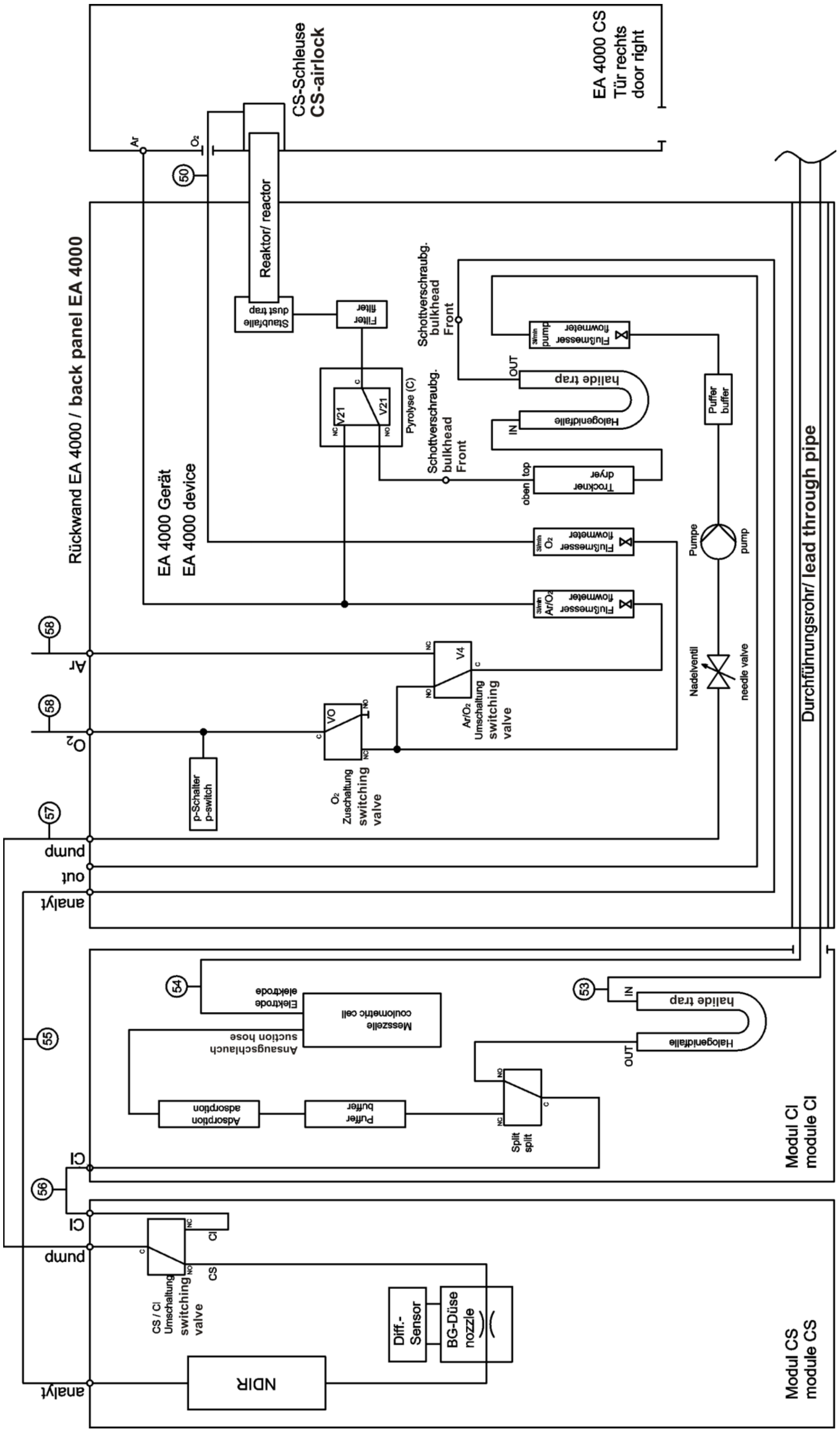


Fig. 39 Hose diagram multi EA C/S Cl for measurement in pyrolysis mode

10.3.4 Installing the automatic TIC solids module

Connections

For the combination of multi EA 4000 – and C/S module - Cl module the following connections must have been made:

multi EA 4000 connections		**Target**
"valve"	⇨	TIC module "valve (HT)"
"analyte"	⇨	TIC module "analyte (HT)"
"O_2"	⇨	Oxygen gas connection
Mains cable	⇨	Mains connection
"COM"	⇨	TIC module "COM (HT)"
"pump"	⇨	C/S module "pump"
"Ar"	⇨	Argon gas connection
"out"	⇨	open output
additional C/S module connections		**Target**
"PC"	⇨	USB connection for PC connection
"valve"	⇨	TIC module "valve (module)"
"scale"	⇨	external scales (optional)
"COM"	⇨	TIC module "COM (module)"
"sampler"	⇨	connection of the sampler FPG 48
"analyte"	⇨	TIC module "analyte (module)" The dust trap must be inserted between the two connection (see Fig. 22 p. 36).
socket for mains connector	⇨	Mains connection
additional connections of the TIC module		**Target**
"waste"	⇨	connection of the waste hose to drain the condensate
socket for mains connector	⇨	Mains connection

Hose diagram

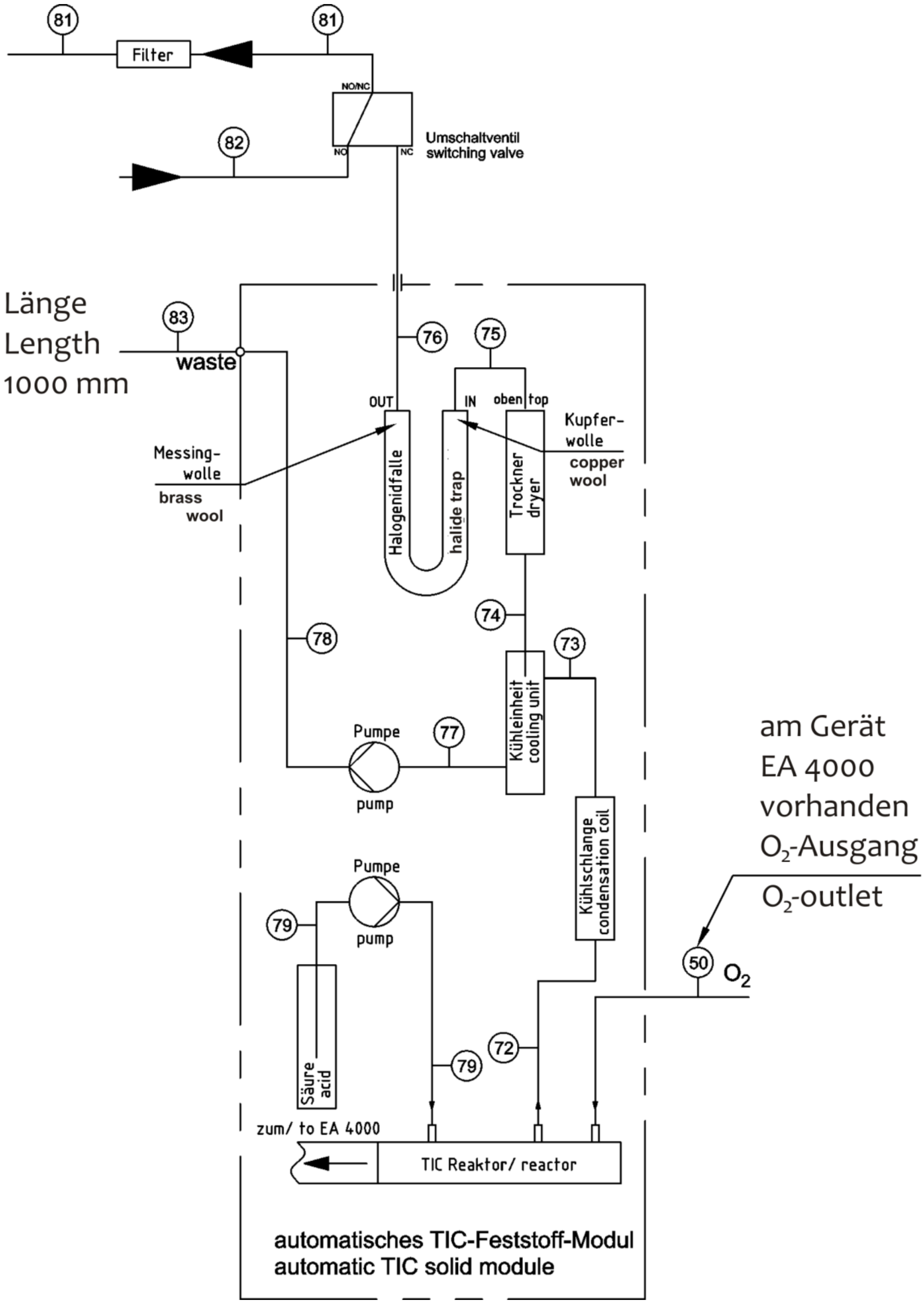


Fig. 40 Hose diagram of the automatic TIC solids module

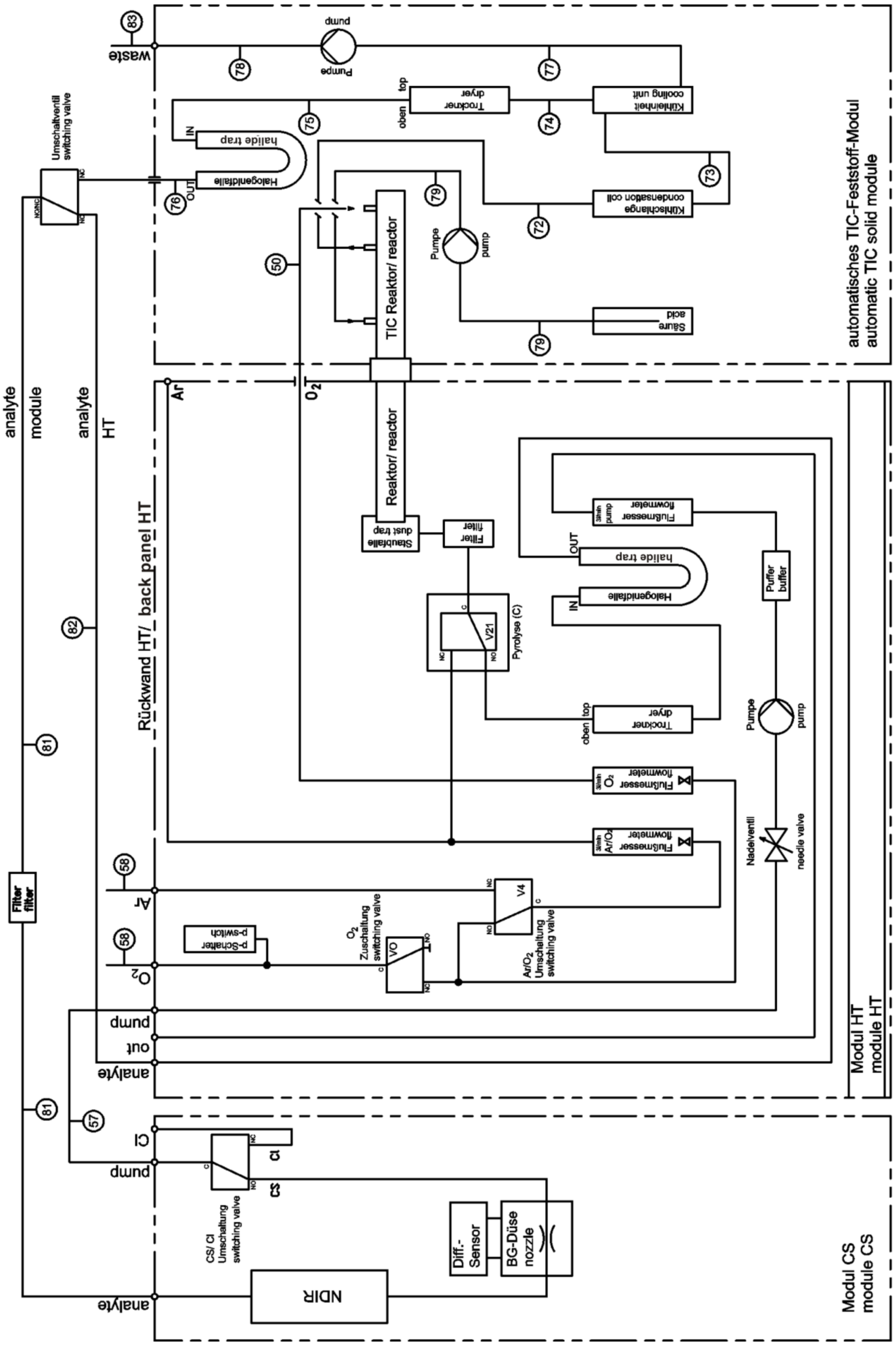


Fig. 41 Hose diagram multi EA C/S with connected automatic TIC solids module

10.3.5 Installing the manual TIC module

Make the connections

For the combination of multi EA 4000 – and C/S module - TIC solids module the following connections must have been made:

multi EA 4000 connections		**Target**
"valve"	⇨	C/S module "valve"
"analyte"	⇨	open
"O_2"	⇨	Oxygen gas connection
Mains cable	⇨	Mains connection
"COM"	⇨	C/S module "COM"
"pump"	⇨	open
"Ar"	⇨	Argon gas connection
"out"	⇨	open output
C/S module connections		**Target**
"PC"	⇨	USB connection for PC connection
"scale"	⇨	external scales (optional)
"pump"	⇨	open
socket for mains connector	⇨	Mains connection
"analyte" connection	⇨	TIC module "analyte"
additional connections of the TIC module		
"O_2"	⇨	oxygen gas connection with quick-release coupling set oxygen system pressure to 2 to 4 bar
socket for mains connector	⇨	Mains connection

Hose diagrams

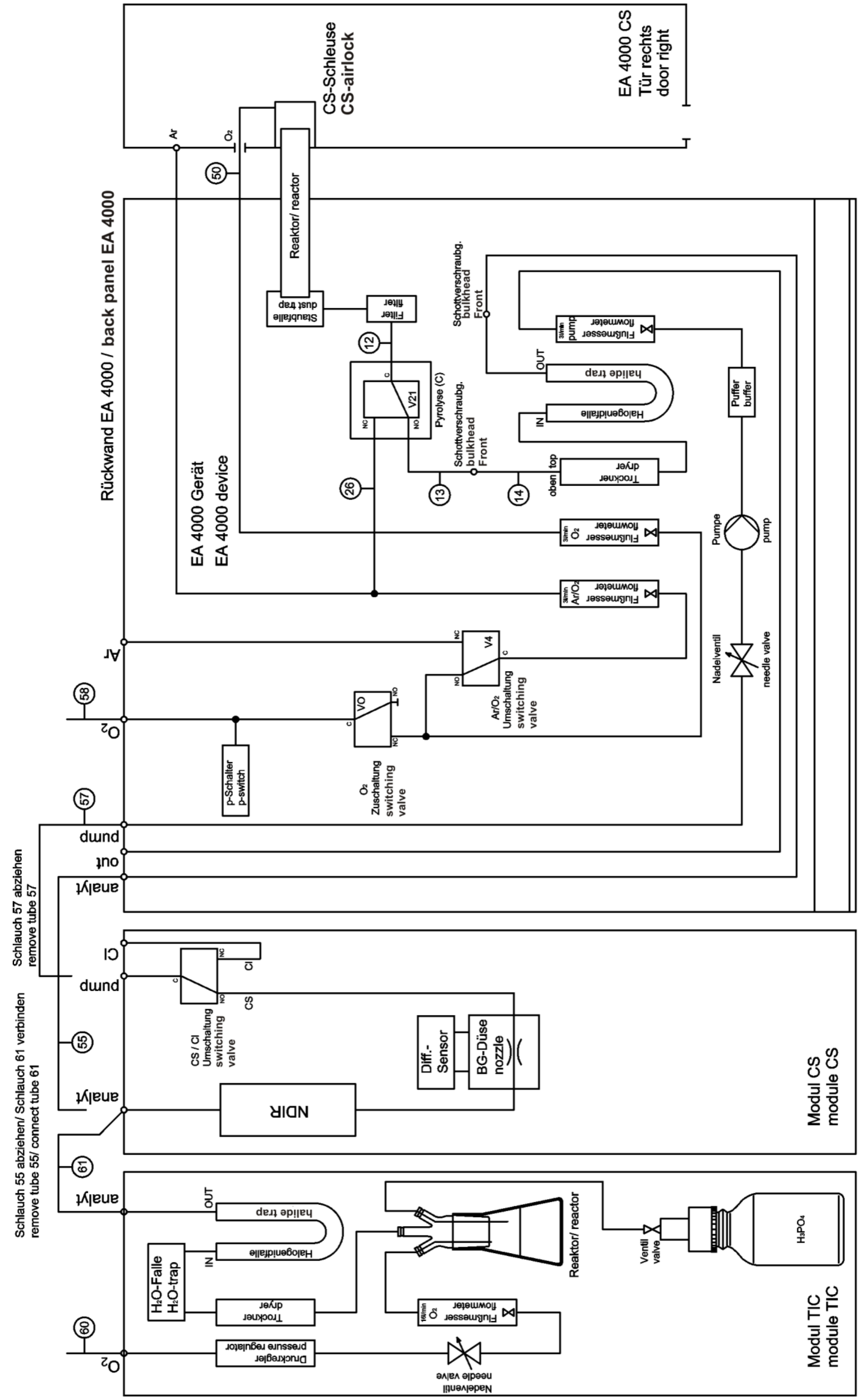


Fig. 42 Hose diagram multi EA C/S with manual TIC solids module

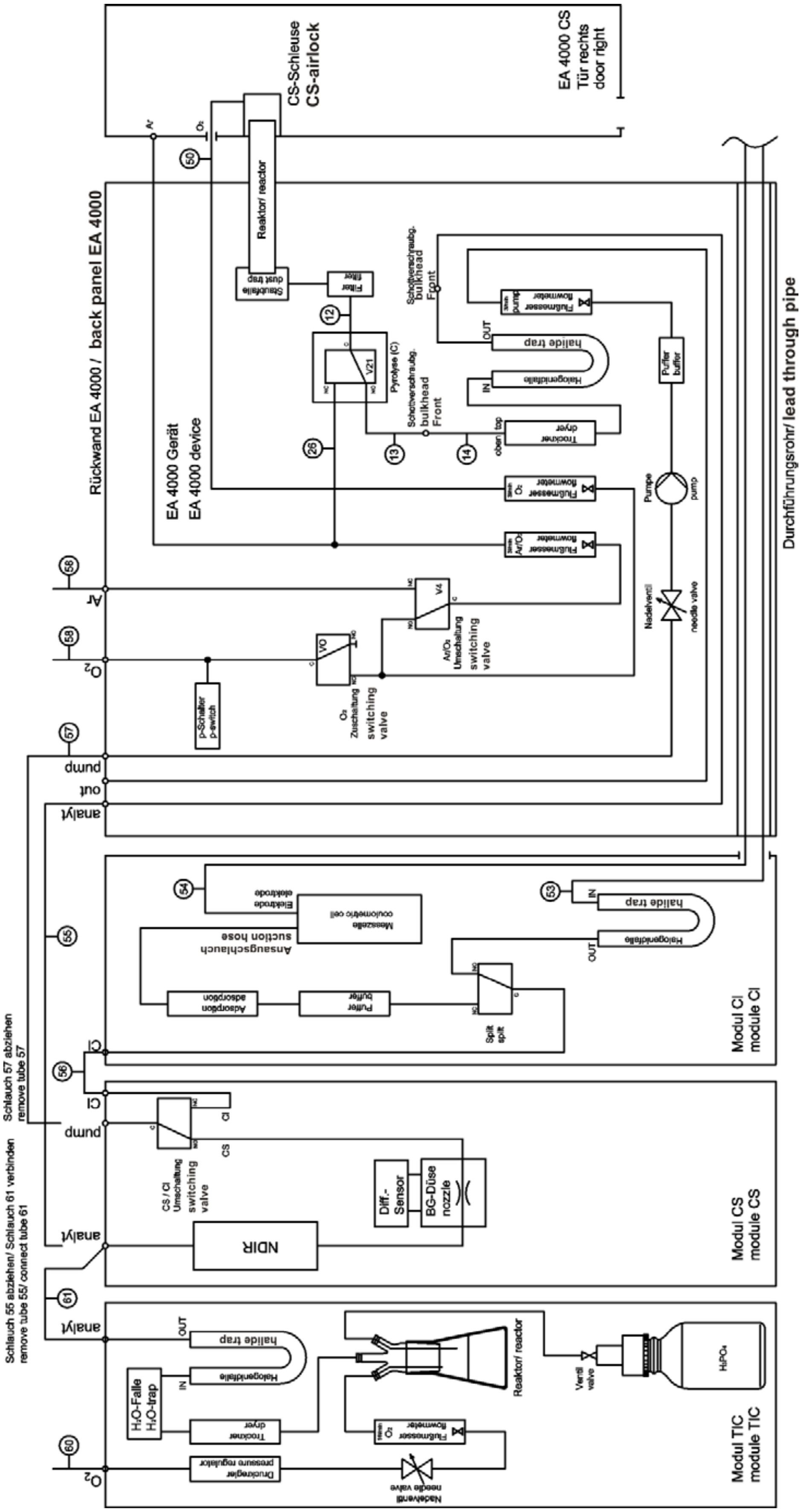


Fig. 43 Hose diagram multi EA C/S Cl with manual TIC solids module

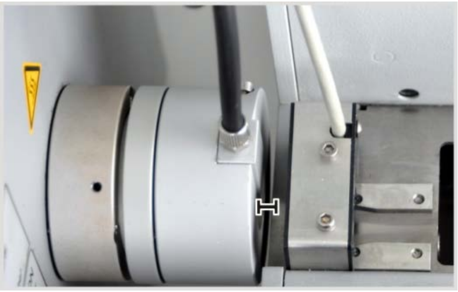
10.4 Connecting and using the sampler

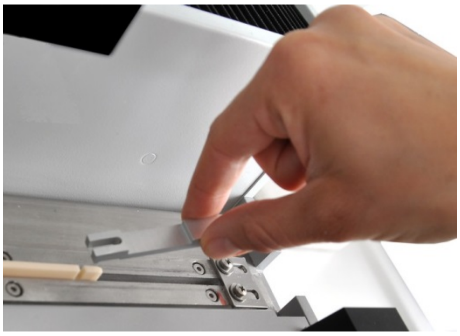
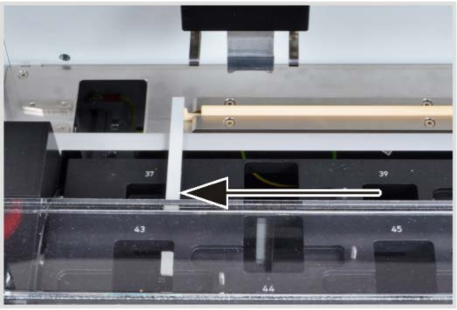
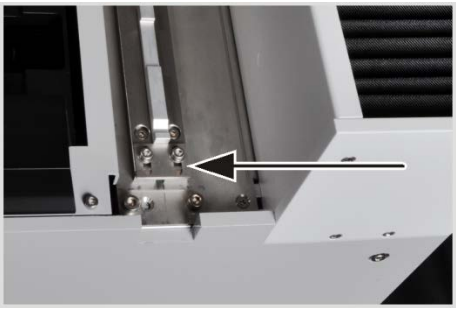
CAUTION

During installation and prior to handling samples make sure that the sampler is parallel to the basic device and level with the operating tube! If the sample boat is moved into the furnace at an angle or not at the correct height relative to the operating tube, the ceramic hook can break and the operating tube can be damaged.

10.4.1 Installing and adjusting the autosampler FPG 48

Connect the sampler to the analyzer as follows:

 	1. Place the sampler to the right of the analyzer. 2. Plug cable on the low voltage side of the table power supply into the connection at the rear of the sampler. Connect the power supply to the mains network. 3. Connect the data cable supplied to the "sampler" interface at the rear of the sampler. Connect the other end of the cable to the "sampler" interface at the rear of the module with the control unit (C/S module or Cl module). 4. Connect the boat sensor to the "aux" connection at the rear of the sampler. 5. Plug the grounding conductor into the connection at the rear of the basic device.
	6. Position the sampler parallel to the basic device. The hook extension must be centered with the gas airlock. **Caution**: A gap of 2 mm must remain between the FPG 48 and the gas airlock to allow for the compensation of thermal expansion of the combustion tube. The FPG 48 must not press against the gas airlock at any time. 7. Using the adjustable feet correct the height of the sampler, if necessary. The bottom edge of the opening of the gas airlock must be level with the upper edge of the storage surface of the sampler.

	8. Place the ceramic hook into the guide rail of the sampler. 9. Place the sliding rod onto the hook until the pin engages in the slot. 10. Place a boat into the feeder (hook the eye of the boat into the hook) and carefully move the boat manually into the furnace. Make sure that the hook is not wedged or angled. Otherwise correct the position of the sampler.
 	Check the start position of the ceramic hook. The hook must be flush with the right-hand edge of the left-hand boat position (here position 37). 11. Place a straight paper or plastic strip (e. g. a ruler) along the edge and align the tip of the ceramic hook with it. 12. If the factory setting is not correct, loosen the screws at the stop plate of the guide rail. Move the plate until it touches the end of the sliding rod. Secure the stop plate with the screws.

Caution!
Do not move the sampler arm manually if the sampler is switched on. The drives of the FPG 48 could be damaged.

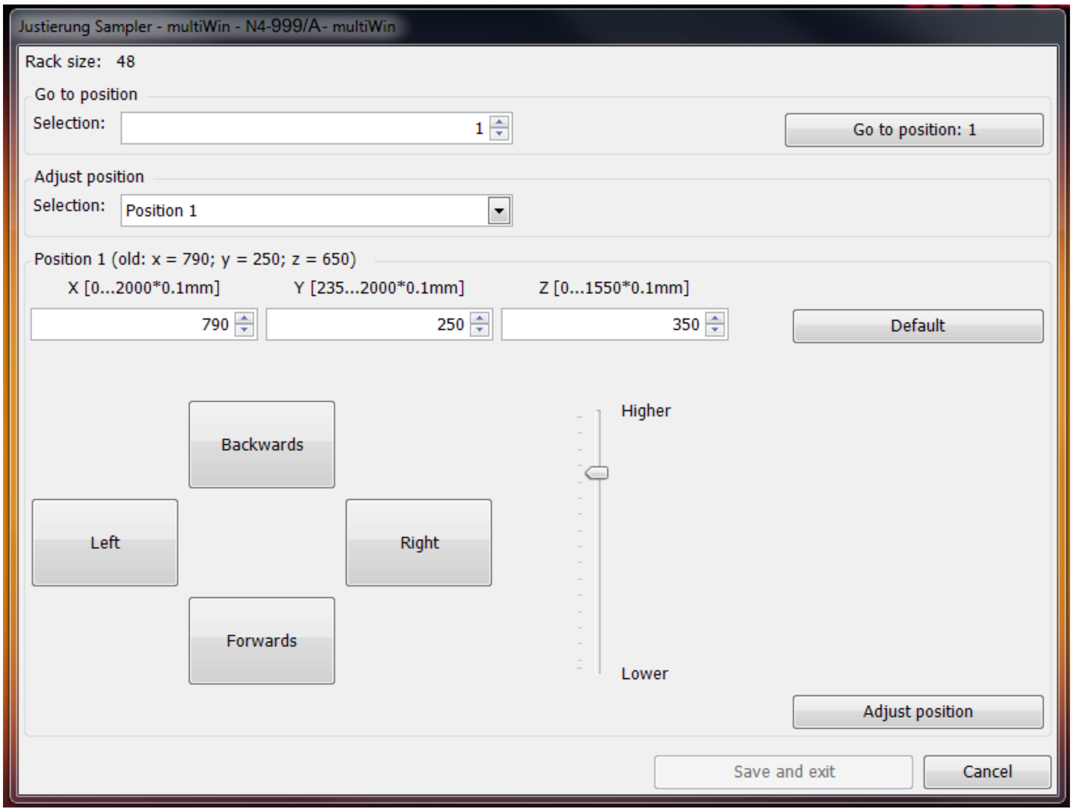
Adjustment of the gripper

Align the gripper using the multiWin software for the following positions:

- ☐ Position 1
- ☐ Sliding position
- ☐ Ejection rail (e. g. after installation of the automatic TIC solids module)

Position 1

1. Switch on the basic device, the detector with control unit, the automatic TIC solids module, where applicable, and the FPG 48. Start the multiWin software and initialize the analyzer.
2. Select the menu command SYSTEM / SAMPLE GENERAL. A window with the same name opens.



Justierung Sampler - multiWin - N4-999/A- multiWin

Rack size: 48

Go to position

Selection: 1

Adjust position

Selection: Position 1

Position 1 (old: x = 790; y = 250; z = 650)

X [0...2000*0.1mm]: 790

Y [235...2000*0.1mm]: 250

Z [0...1550*0.1mm]: 350

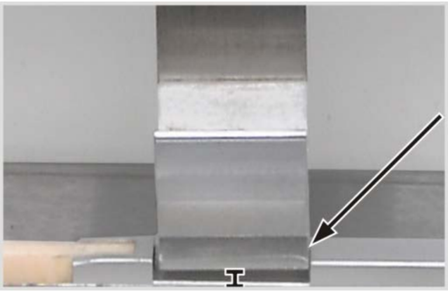
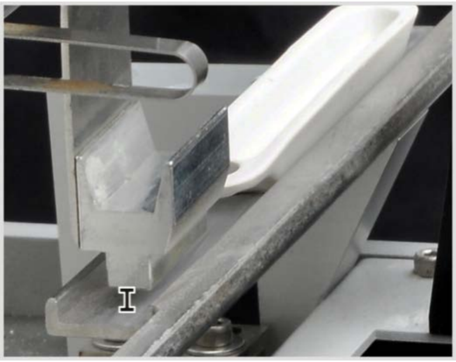
3. Place the white adjustment aid into the boat position to be adjusted on the rack (here position 37).
4. Select the option Position 1 from the list and click on [Adjust Position]. The sampler arm moves above the left-hand boat position.
5. Check that the gripper is centered above the opening of the adjustment aid and can enter the opening without obstruction. Correct the alignment in the x and y directions using the arrow keys where necessary.

   For rough adjustment select a z value of 350, for the fine adjustment values from 450-560. This prevents the gripper from impacting hard with the sampler.

   Check the changes by clicking on [Adjust Position].

6. Adjust the lowering depth of the gripper.

   Click again on [Adjust Position] and remove the adjustment aid during the return of the sampler arm to the start position.
7. Using the slider Higher / Lower adjust the lowering depth of the gripper and set the new position by clicking on [Adjust Position].
8. Repeat the process until the gripper is positioned just a few millimeters below the boat storage.

	This prevents the boat from moving when picked up by the gripper. 9. Click on [SAVE AND EXIT].
	**Sliding position** The sliding position is the cut-out in the sliding rod of the ceramic hook. The sampler arm enters this cut-out and moves the hook into the furnace. 1. Place the ceramic hook and sliding rod against the stop plate of the FPG 48. 2. Select the option SLIDING POSITION in the SAMPLER ALIGNMENT window from the list and click on [ADJUST POSITION]. The sampler arm moves above the sliding position. 3. Using the slider HIGHER / LOWER adjust the lowering depth of the gripper. Using the buttons [LEFT] / [RIGHT] and [BACKWARDS] / [FORWARDS] adjust the position in the cut-out. The gripper must be adjusted flush right and centered in the cut-out. The gripper must hover approx. 1 mm above the cut-out. The sampler arm must not exert any pressure on the sliding rod. 4. Set the new position by clicking on [ADJUST POSITION]. 5. Repeat the process until the adjustment is correct. Then click on [SAVE AND EXIT].
	**Ejection rail** Only if the boat storage is fitted (e.g. when using the automatic TIC solids module) 1. Select the option EJECTION RAIL from the list in the SAMPLER ALIGNMENT window and click on [ADJUST POSITION]. 2. Using the slider HIGHER / LOWER adjust the lowering depth of the gripper. The gripper must hover approx. 1 mm above the rail and must not rest on it. 3. Place a boat next to the gripper and check that the boat cannot slide under the gripper. 4. Set the new position by clicking on [ADJUST POSITION]. 5. Once the position has been adjusted click on [SAVE AND EXIT].

10.4.2 Filling the sampler FPG 48

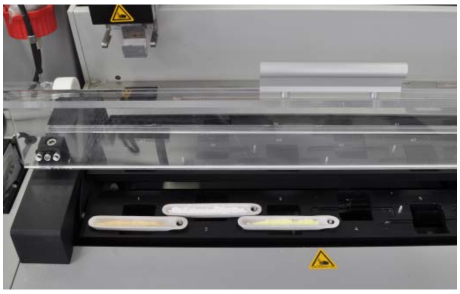
CAUTION! RISK OF CRUSHING!

The rotating sample rack and the moving boat gripper can crush your hand.

- Great caution is required when filling the sample rack.
- After filling, close the acrylic glass cover above the sample rack.
- Do not reach into the sample rack or the boat gripper during an ongoing measurement.

The FPG 48 is filled during the measurement preparation for the multi EA 4000. The multi EA 4000 and the FPG 48 must be switched on and have been initialized in the multiWin program.

6. Load sample material into the ceramic boats and make the boats available adjacent to the sampler.
7. Fold the cover above the sample rack upwards.
8. Using the buttons ↻ and ↷ in multiWin rotate the sample rack into the desired position.



9. Place the loaded boats into the corresponding sample positions. The short metal pin to the right of the sample position must protrude into the opening of the boat.
10. After filling the sample rack fold the cover down.

10.4.3 Measurement cancellation when using the sampler FPG 48

After a manual measurement cancellation or a measurement cancellation due to device fault may result in the ceramic hook being in an unfavorable location and breaking off if the measurement is continued.

- Move the ceramic hook manually to the limit stop before resuming the measurement process.

10.4.4 Retrofitting the sampler FPG 48 with a boat sensor

The boat sensor monitors the boat transfer from the sampler FPG 48 to the furnace. It detects the following fault conditions:

- Ceramic boat broken
- No ceramic boat on the hook

If one of the fault conditions occurs, the measurement is canceled immediately. An error message is issued in the multiWin software.

Note: The boat sensor can also be ordered separately and retrofitted.

Install the boat sensor on the sampler as follows:

	1. Slide the boat sensor from the left onto the short guide rail of the FPG 48. 2. Using the two hexagon socket screws tighten the boat sensor. 3. Connect the boat sensor to the "aux" connection at the rear of the FPG 48.

To resume a measurement after cancellation:

1. Confirm the error in the software.
2. Remove the broken boat, if applicable.
3. Slide the ceramic hook and its sliding rod manually to the stop plate.
4. Re-initialize the device.

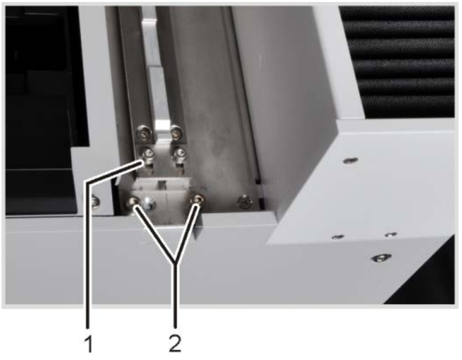
10.4.5 Retrofitting the sampler FPG 48 with a boat storage

For measurements using the automatic TIC solids module the sampler must be converted. An ejection rail for used boats and a cover for the longer ceramic rod are retrofitted.

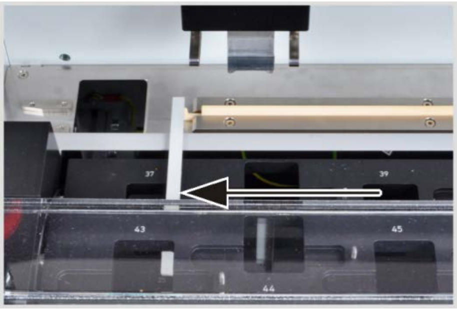
Note: The boat storage can also be ordered separately and retrofitted.

1	block with ejection rail
2	hexagon socket screws with washers
3	cover for ceramic hook

Fig. 44 **Retrofit kit for FPG 48 to use the boat storage**

	1. Unscrew the stop plate of the guide rail (1). 2. Remove the two hexagon socket screws (2).

3. Using the screws supplied screw the block with the boat storage loosely to the FPG 48.
4. Slide the hook cover under the block.



5. Place the ceramic hook into the guide rail of the sampler.
6. Place the sliding rod onto the hook until the pin engages in the slot.
7. Adjust the ceramic hook in the start position. The hook tip must be flush with the right-hand edge of the left-hand boat position (here position 37).

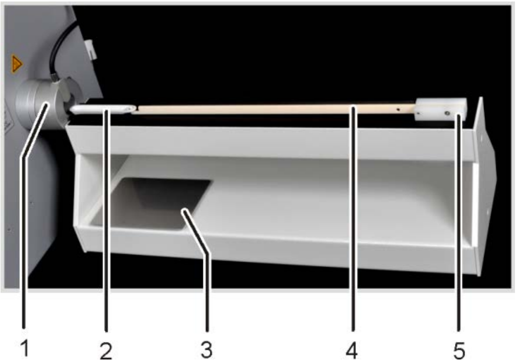
   Place a straight paper or plastic strip (e. g. a ruler) along the edge and align the ceramic hook with it.

8. Move the cover until the rear end of the sliding rod touches the stop plate at the end of the cover. (see image section for stop plate)
9. Tighten the hexagon socket screws at the block finger-tight and thereby clamp the cover in its adjusted position.
10. Start the multiWin software and log in as administrator.
11. Select the menu command DEVICE / DEVICE – EDIT.

    A window with the same name opens.
12. In the area ACCESSORY MODULES place a tick for EJECTION RAIL.
13. Adjust the position of the gripper relative to the ejection rail (see section "Installing and adjusting the autosampler FPG 48" p. 137).

10.4.6 Using the manual sample feed

Install the manual sample feed as follows:



1	gas airlock	4	ceramic hook
2	ceramic boat	5	handle
3	storage area		

1. Position the manual sample feed in front of the gas airlock. Align the sample feed until the ceramic boat is moved exactly centered into the gas airlock.
   The distance between the sample feed and the gas airlock should be approx. 2 mm.
2. Place a boat with its eye on the hook of the sample feed and move the boat into the furnace.

   Caution:
   Only hold the hook from the handle. There is a risk of burns at the hot furnace.

   Make sure that the boat does not get wedged, otherwise correct the position of the sample feed.
3. Using pliers remove the hot boats from the hook and place them onto the storage area to cool down.

10.5 Connecting and using the external scales

When using external scales the sample weights can be transferred directly into the sequence table.

1. Connect the scales to the "scale" connection of the C/S module or Cl module.
2. Connect the scales to the mains supply.
3. Set the baud rate to 9600.
4. For additional configurations refer to the documentation of your scales.

To transfer the sample weights proceed as follows:

1. Start the measurement up to the entry of the analysis sequence.
2. In the sequence table click on the field WEIGHT of the sample.
3. Weigh the sample on boat.
4. Accept the value from the scales with [OK].

   ✓ **The weighted sample appears in the input field.**
5. Repeat steps (2) - (4) for each sequence table.

11 Disposal

ENVIRONMENTAL PROTECTION

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

Sulfuric acid

The used desiccant from the Cl detection is first carefully diluted with water and then neutralized with caustic soda. The corresponding hazard notices and safety notes must be observed! This neutralized waste must be disposed of correctly in accordance with the statutory requirements.

Magnesium perchlorate

The used desiccant from the C and S detection is converted into less hazardous reaction products by adding a sodium sulfate solution – where necessary with added acid. This neutralized (where applicable) waste must be disposed of correctly in accordance with the statutory requirements.

Electrolyte solution

The electrolyte solution is carefully neutralized and collected as silver salt residue in containers. This must be disposed of or recycled correctly in accordance with the statutory requirements.

Adsorption agent

The active carbon used for the adsorption of acetic acid vapor must be disposed of in accordance with the public regulations.

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.

Analyzer

At the end of its service life the multi EA 4000 and all its electronic components must be disposed of as electronic waste in accordance with the applicable regulations.