

Operating Manual

ICprep

Pyrohydrolytic digestion system



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

Technical Service Analytik Jena GmbH+Co. KG
Konrad-Zuse-Straße 1
07745 Jena / Germany
Phone: +49 3641 77 7407
Fax: +49 3641 77 9279
Email: service@analytik-jena.com



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

General Information <http://www.analytik-jena.com>

Documentation Number 11-0608-001-23

Edition A (05/2025)

Technical Documentation Analytik Jena GmbH+Co. KG

© Copyright 2025, Analytik Jena GmbH+Co. KG

Table of contents

1 Basic information	5
1.1 About this manual	5
1.2 Intended use	6
2 Security	7
2.1 Safety labeling on the device	7
2.2 Requirements for the operating personnel.....	8
2.3 Safety instructions for transport and commissioning.....	8
2.4 Safety instructions for operation	9
2.5 Safety instructions for the operation of compressed gas containers and compressed gas systems.....	10
2.6 Safety instructions for maintenance and cleaning	10
2.7 Behavior during emergencies	11
3 Function and design	12
3.1 Structure of the ICprep digestion system	12
3.1.1 Base module	12
3.1.2 Dosing modules	18
3.1.3 Automatic Boat Drive (ABD)	20
3.1.4 Multi Matrix Sampler	21
3.1.5 Fraction collector for automated operation	21
3.1.6 Type plates	23
3.2 Principle of operation	23
4 Installation and commissioning	24
4.1 Installation conditions.....	24
4.1.1 Installation location requirements.....	24
4.1.2 Power supply.....	24
4.1.3 Gas supply	24
4.1.4 Device layout and space requirements.....	24
4.2 Unpacking and setting up the device.....	25
4.2.1 Installing the digestion system	26
4.2.2 Removing and installing the combustion furnace	27
4.2.3 Installing dosing modules and transfer line.....	28
4.2.4 Removing and installing the Peltier cooling block.....	31
4.2.5 Installing the ABD.....	31
4.2.6 Installing the fraction collector	33
4.2.7 Installing the ceramic combustion tube	36
4.3 Converting multi EA 5100 or multi X 2500.....	38
4.4 Software installation and device setup.....	39
5 Operation.....	41
5.1 General information on sample digestion.....	41
5.2 Switching on the digestion system	41
5.3 Switching off the digestion system.....	42
5.4 Settings in the multiWin software.....	42
5.4.1 Create methods.....	43
5.4.2 Creating an analysis sequence	46

5.4.3	Create a transport program for the ABD	49
5.5	Start digestion	50
5.6	Sample feed for the digestion system	50
5.6.1	Prepare the quartz boats for the sample feed	50
5.6.2	Sample feed in automatic operating mode	51
5.6.3	Sample feed in manual operating mode	53
6	Maintenance and care	55
6.1	Maintenance intervals	55
6.2	Cleaning the device	56
6.3	Maintenance of the base module	56
6.3.1	Removing and cleaning the combustion tube	56
6.3.2	Installing the combustion tube	57
6.3.3	Replacing the hose connections	58
6.3.4	Remove and clean the T-connector	60
6.3.5	Replacing the check valves on the gas box	61
6.3.6	Replacing the particle filters in the gas inlets	62
6.3.7	Maintenance of the dosing syringes	63
6.3.8	Replacing tubes	64
6.4	Maintenance of the ABD	66
6.4.1	Disconnecting the ABD from the combustion tube	66
6.4.2	Installing the ABD at the combustion tube	67
6.4.3	Replacing the septum and cleaning the sluice at the ABD	68
6.4.4	Replacing the seal on the sample port	68
6.4.5	Cleaning and changing hooks	69
6.4.6	Cleaning or replacing the guide tube	70
6.5	Checking the system for leaks	71
6.6	Recommissioning after emergency shutdown or shutdown due to gas pressure fault	73
7	Troubleshooting	75
7.1	Remedying software message faults	75
7.2	Initializing the basic module and the system components	78
7.3	Device faults on the basic module	78
7.4	Analytical problems in the basic module	79
8	Transport and storage	81
8.1	Transport	81
8.1.1	Moving the device in the laboratory	81
8.1.2	Preparing the device for transportation and storage	81
8.2	Storage	84
9	Specifications	85
9.1	Technical data for the ICprep	85
9.2	Guidelines and standards for ICprep dosing modules	86

1 Basic information

1.1 About this manual

The operating manual describes the following digestion system:

- ICprep

Applicable documents

The digestion system is controlled via the multiWin software. An ABD is used for the sample feed. For the automated version of the ICprep automatic digestion system, the system is fitted with a Multi Matrix Sampler autosampler and a fraction collector. These instructions apply with the following documents:

- Automatic Boat Drive (ABD) operating manual
- Multi Matrix Sampler operating manual
- multiWin operating manual

The device is intended to be operated by qualified specialist personnel who must observe all instructions given in the operating manual.

The operating manual provides information about the design and operation of the device and provides operating personnel with the necessary know-how for safe handling of the device and its components. Furthermore, the operating manual includes information on the maintenance and servicing of the device as well as information on potential causes of malfunctions and their correction.

Conventions

Instructions for actions are marked with a black triangle (►). Results of actions are marked with a checkmark (✓).

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

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

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

Symbols and signal words used in this manual

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



WARNING

Indicates a potentially hazardous situation which can cause death or very serious injuries with permanent damage.



CAUTION

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



NOTICE

Provides information on potential material or environmental damage.

1.2 Intended use

The ICprep digestion system is suitable for solid and liquid organic samples whose halogen content (F, Cl, Br, I) or sulfur content is to be determined after digestion using ion chromatography or other suitable detectors (e.g., ISE, MAS, photometer). Digestion is carried out by pyrohydrolysis with subsequent thermal oxidation.

The digestion system and connected components are controlled via the multiWin control and analysis software.

The digestion system may only be used for the applications described in this user manual. Only this specified use is regarded as the intended use, ensuring the safety of the user and the device.

Samples suitable for ICprep	■ Samples for determining the sum parameters AOF, AOCl, AOBr, AOJ, ClC-AOX The samples can be enriched using the column method on Analytik Jena columns (18 x 6 mm columns) or using the batch method with filtration on polycarbonate filters. ■ Samples for the determination of EOF ■ Organic solids, e.g. polymers ■ Organic liquids, e.g. solvents ■ Highly viscous organic liquids, gels and paste samples (e.g. pyrolysis oil)
Samples that are not suitable	The following samples must not be processed with the ICprep digestion module: ■ Highly flammable organic compounds – explosion hazard ■ Self-reactive materials and explosives, e.g., peroxides, epoxides, azides ■ Organic and inorganic phosphorous compounds, e.g., phosphoric acid ester ■ Organosilicon and reactive or unstable inorganic silicon compounds, e.g. silanes ■ Metal organic compounds, e.g., nickel carbonyl ■ Corrosive or highly-reactive substances, inorganic acids, HF, Cl₂, H₂SO₄ ■ Inorganic compounds, e.g., minerals, inorganic halogens ■ Samples that require more than 1100 °C for complete oxidation or decomposition, e.g. metals, alloys
Further notes on use	Alkaline and alkaline earth salts cause crystallization in all quartz glass components. An optional ceramic combustion tube is available for saline samples. Depending on the initial fluoride concentration in the sample and the water input, different HF concentrations can be expected in the digested sample. From 30% fluoride in the initial sample, HF concentrations of > 1% can occur in the digested sample after digestion (correspondingly higher depending on the sample composition). It is recommended to work at 1050 °C. Higher digestion temperatures lead to faster wear of the combustion tube and the furnace.
Solutions used for dosing modules	Only aqueous solutions, e.g. ultrapure water and hydrogen peroxide solution, may be dosed with the dosing modules. No flammable solutions or substances that can form explosive mixtures may be dosed.
Gases used	The digestion system must only be used with argon and oxygen of the required quality as a carrier gas.

2 Security

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

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

2.1 Safety labeling on the device

Warning and mandatory action labels have been attached to the device and must always be observed.

Damaged or missing warning and mandatory action labels can cause incorrect actions leading to personal injury or material damage. The labels must not be removed. Damaged warning and mandatory action labels must be replaced immediately!

The following warning and mandatory action labels have been attached to the device:

Warning symbol	Meaning	Comment
	Warning against dangerous electrical voltage	In the device interior on the electronics cover Before opening the electronics the device must be disconnected from the mains.
	Warning against hot surface	In the device interior on the combustion furnace On the top cover of the combustion furnace On the heated transfer line for the water supply Touching the furnace or the ends of the transfer line during operation or shortly after switching off the device can cause burns.
	Warning of crushing	On the dosing modules: There is a risk of injury to fingers if you reach into the syringe slot while the pump is running. On the MMS autosampler and fraction collector: There is a risk of crushing in the movement range of the autosampler.
	Warning of sharp objects	On the MMS autosampler and fraction collector: There is a risk of puncture injuries on the autosampler cannula.
	Warning against corrosive substances	On the plate of the fraction collector: When used as intended, the digested sample solutions contain hydrofluoric acid and other corrosive acids, e.g. hydrochloric acid, sulphuric acid.
	Warning of toxic substances	

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

2.2 Requirements for the operating personnel

The device must only be operated by qualified specialist personnel instructed in the use of the device. The operating personnel must meet the following requirements:

- Operate the device only after instruction and training.
- Know and avoid dangers when working with the device.
- Wear personal protective equipment such as protective gloves, lab coat and safety goggles.
- Training by Analytik Jena is recommended.

The responsible body at the installation site is accountable for compliance with safety and occupational health regulations. The responsible body must meet the following requirements:

- Provide information about national regulations on work safety and accident prevention and observe them during operation of the device.
- Instruct the operating personnel in the safe operation of the device. In doing so, also convey the contents of the manuals for the device system.

2.3 Safety instructions for transport and commissioning

Transport

The device is heavy. There is a risk of injury when lifting and carrying, especially from unsecured parts.

- Empty the device. Secure the device components according to the instructions. Remove loose parts and pack separately.
- Only lift the device with two people. Position on opposite sides of the device and grasp the underside of the device firmly with both hands.
- Only transport the device in its original packaging. Insert all transport locks.
- Decontaminate the device before returning it to the manufacturer. Document the cleaning measures in the decontamination record. The decontamination record is provided by customer service when a return is registered.

Ambient conditions during commissioning

The device is dangerous if it is installed in an unsuitable environment. If the device is installed in an unsuitable environment, its service life will be reduced, e.g., due to corrosion.

- Design the installation site in accordance with the requirements in the installation conditions with installation plan.
- The device must not be installed in an explosive environment.
- Ensure free access to the main switch on the rear of the device.
- Keep the ventilation slits clear.

- Depending on the initial sample, hydrofluoric acid is produced in the digestion process. Ventilate the operating area sufficiently. Use a suitable workplace extraction system.

Electrical conditions

The device may be dangerous if the conditions for the electrical connection are not met.

- Only the Analytik Jena customer service or trained and authorized specialist personnel are allowed to install and commission the device and its system components. Unauthorized assembly and installation is not permitted.
- Only use the supplied power cable or a cable of the same size with a protective earth conductor. Do not use an extension in the supply cable.
- Connect the power plug to a proper power outlet to ensure that the device meets protection class I (ground connector). Do not invalidate the protective effect by the use of an extension line which does not have a protective conductor.
- Check the electrical requirements of the device before connecting it to the mains.
- Only connect the device and its system components to the power grid when they are switched off.
- Only connect and disconnect the connection cables between the device and its system components when the device is switched off.

2.4 Safety instructions for operation

Electrical hazard

Lethal voltages may occur in the device.

- Before each start-up, make sure that the device and its safety devices are in proper working condition.
- In case of malfunctions of electrical components, switch off the device immediately and disconnect it from the electrical power supply.
- Do not remove or bypass any protective devices such as the housing.
- Prevent liquid from penetrating the device.

Thermal hazard

The combustion furnace operates at temperatures of up to 1100 °C. There is a risk of burns on the furnace and the combustion tube.

- Do not touch hot parts during or immediately after operation.

Mechanical hazard

During operation, there is a risk of crushing on the moving parts of the syringe pumps and a risk of injury at the autosampler and fraction collector cannulas.

- Maintain a safe distance from moving parts.

The glass parts can break. There is a risk of injury from broken glass and glass splinters.

- Handle glass parts with care.

Hazard from substances

The device can be used to handle hazardous substances. The responsible body at the installation site is accountable for the safe handling and disposal of hazardous substances.

- Depending on the initial sample, hydrofluoric acid and other corrosive acids, e.g. hydrochloric acid, sulphuric acid, are produced in the digestion process. Wear suitable protective clothing. Ensure adequate room ventilation, e.g. by means of a suitable workplace extraction system.
- The digestion system may only be used for the samples specified in the "Intended use" section.
- Only aqueous solutions may be used with the dosing modules. No flammable liquids or substances that can form explosive mixtures may be dosed.
- Only use dosing modules with the safety tray fitted. Ensure that the tubing is securely seated.
- When working with samples dissolved in organic solvents, take the appropriate fire safety and health protection measures.

- If the device has been contaminated with hazardous substances, decontaminate it as described in the operating manual. Before using a cleaning or decontamination procedure other than that prescribed by the manufacturer, the user is required to check with Analytik Jena that the intended procedure will not damage the device.

2.5 Safety instructions for the operation of compressed gas containers and compressed gas systems

The operating gases are taken from compressed gas containers or local compressed gas systems. The responsible body at the installation site is in charge of ensuring the safety of the gas system.

For safe operation, pay particular attention to the following points: Any leaks in gas systems and pipes, except for oxygen and air, can lead to an oxygen-deficient atmosphere. There is a risk of suffocation in unventilated rooms.

If oxygen accumulates in the atmosphere, flammable substances can ignite very easily and burn violently. Contact between oil or grease and oxygen under high pressure can cause explosions.

- Work on pressurized gas vessels and systems must only be carried out by trained and competent personnel. Do not carry out any unauthorized assembly or installation work.
- Only use pressure hoses and pressure reducers for the gases for which they are intended.
- Keep pipes, hoses, screw connections, and pressure reducers for oxygen free of oil and grease.
- Inspect all pipes, hoses, and screw connections regularly for leaks and damage. Repair leaks and damage without delay.
- Shut off the gas supply to the device before working on the pressurized gas vessels or the pressurized gas system. Only restart the device after completing the work and checking that it is functioning properly.

2.6 Safety instructions for maintenance and cleaning

There is a risk of electric shock if contact is made with live components, which may lead to serious injury.

Unauthorized servicing can lead to maladjustment or damage of the device and its system components.

- Work on electrical components inside the device may be carried out by customer service only.
- Only carry out the maintenance actions listed in the operating manual.
- Switch off the device before maintenance and cleaning. Only work on a switched-on device if this is expressly required by the operating manual.
- Before performing maintenance work inside the device, disconnect the power plug from the power outlet.
- Shut off the gas supply to the device before performing maintenance. Only leave the gas supply open if expressly required by the operating instructions.
- Use only original spare parts, wear parts and consumables. These have been tested and ensure safe operation.
- After maintenance, ensure that all safety devices are fully functional again.
- Clean the device with a damp, non-dripping cloth. Do not use organic solvents, abrasive cleaners or bleach.

2.7 Behavior during emergencies

- In dangerous situations or in the event of accidents, immediately press the "Stop" button on the top of the ABD.
- If there is no immediate risk of injury, switch off the device and its system components at the mains switches and then pull the mains plugs out of the mains sockets.
- After switching off the device, close the gas supply.

3 Function and design

3.1 Structure of the ICprep digestion system

The digestion system is supplied in two model variants. The ICprep basic model for manual operation consists of the following components:

- Base module with combustion furnace
- Dosing modules for the supply of ultrapure water and absorber solution, as well as waste collection during system purging
- ABD for sample supply to the combustion tube
- Stand for cannula holder and rack for sample tubes to collect the fractions

The ICprep automatic model for automatic operation includes the MMS autosampler and fraction collector in addition to the components listed above.

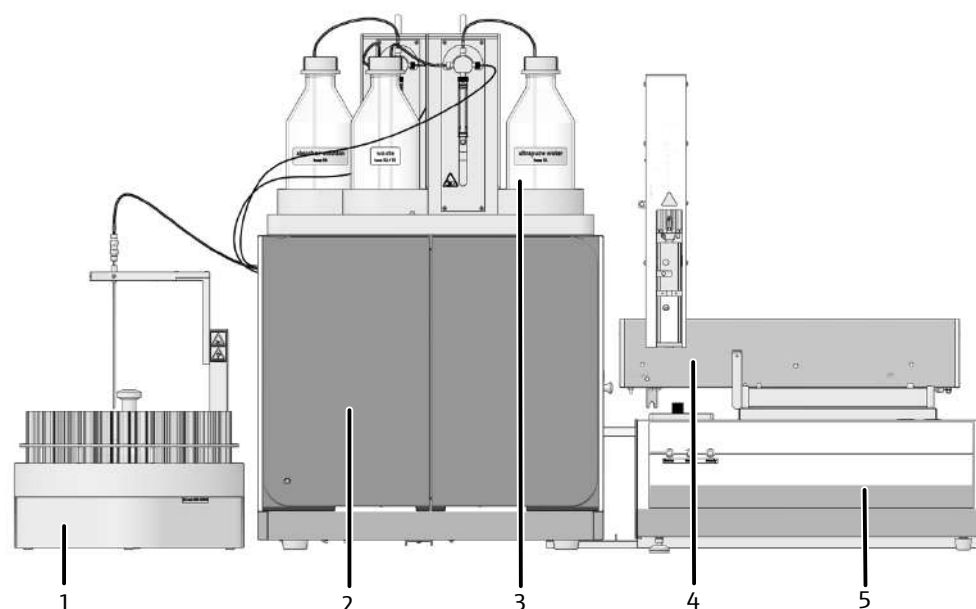


Fig. 1 Model ICprep automatic

- | | |
|----------------------|---------------|
| 1 Fraction collector | 2 Base module |
| 3 Dosing modules | 4 MMS |
| 5 ABD | |

A self-check system (SCS) is integrated into the digestion system. The SCS is a combination of hardware components and software functions that independently ensure the error-free operation of the entire device system. The SCS is used to check the parameters that are important for device safety and digestion quality (e.g. gas flows, temperatures, pressures, cooling time and flame values) several times a second.

3.1.1 Base module

The base module contains the following components:

- Electronics and device control
- Combustion system
- Cooling of the digested samples and sample transfer

All components that require user maintenance are accessible via the front doors and the removable side panels.

Electrical connections, connections for the gas supply and interfaces for other system components are located on the rear of the base module.

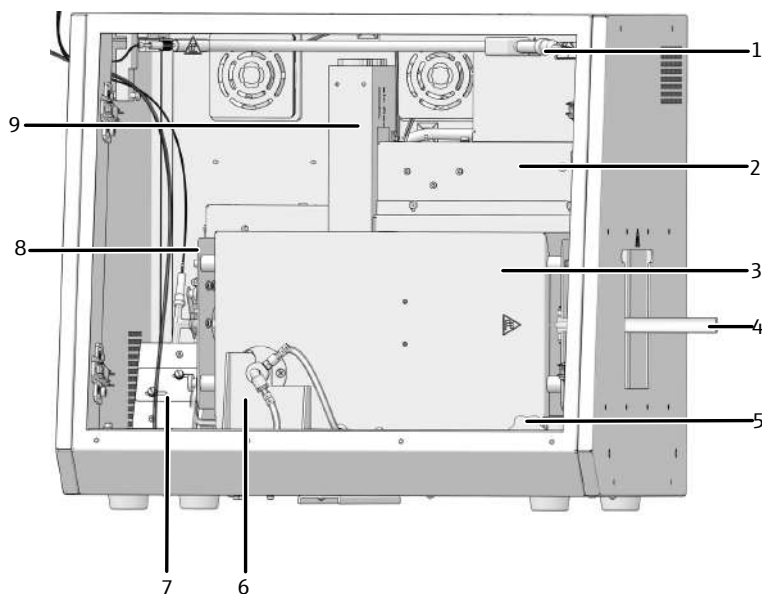


Fig. 2 Base module (without front doors)

- | | |
|--|----------------------------|
| 1 Heated transfer line for ultrapure water | 2 Control electronics |
| 3 Combustion furnace | 4 Combustion tube |
| 5 Knob for turning the furnace | 6 Furnace mounting |
| 7 Peltier cooling block with T-connector | 8 Gas supply (not visible) |
| 9 Power supply unit for the combustion furnace | |

3.1.1.1 Electrical components and connections

Internal device control

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

Operational LED

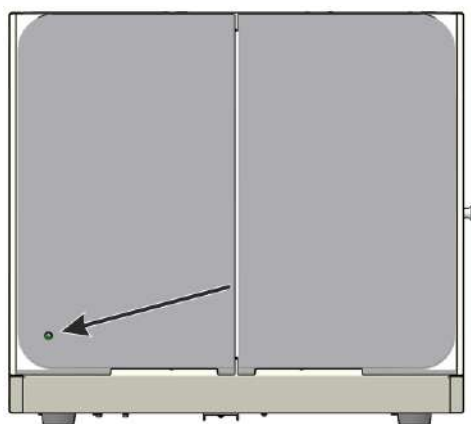


Fig. 3 LED display for operational readiness

A green LED is installed on the left door of the base module. After device initialization, i.e., connection between device and multiWin PC/software, the LED lights up and indicates that the device is ready for operation.

Mains switch, interfaces and gas connections

The mains switch and the interfaces for connecting the system modules, as well as the PC connection are located on the rear of the device. The interfaces for the autosampler and dosing modules are RS232 interfaces.

The gas connections are also located on the rear of the device

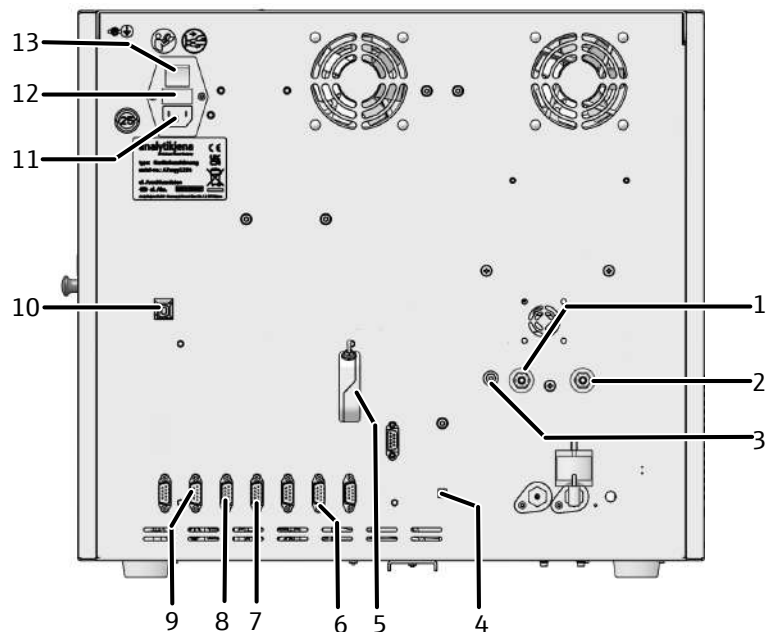


Fig. 4 Connections on the rear of the device

- | | |
|---|---|
| 1 "IN O2" oxygen gas input | 2 "IN Ar" argon gas input |
| 3 "OUT ABD" ABD gas connection | 4 "flame" connection of flame sensor to control the ABD |
| 5 "external" dangle for switching between analyzer and ICprep operating modes | 6 "S-UVF" fraction collector connection |
| 7 "N-CLD" absorber module connection | 8 "C-NDIR" humidifier module connection |
| 9 "sampler" ABD or MMS 5100 connection | 10 "PC-USB" USB connection for PC |
| 11 Power supply | 12 Fuse holder |
| 13 Power switch | |

Interfaces in the device

The electrical connections for the combustion furnace, the flame sensor and the temperature sensor are located on the inside rear of the device. The connections are only accessible when the combustion furnace is installed vertically.

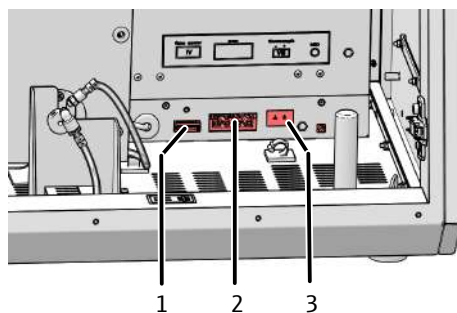


Fig. 5 Electrical connections in the interior of the base module

- | | |
|----------------------|----------------------|
| 1 Flame sensor | 2 Combustion furnace |
| 3 Temperature sensor | |

The connection of the heated transfer line for the water supply to the combustion tube is located on the frame behind the door.



Fig. 6 Electrical connection for the heated transfer line

The electrical connection for the Peltier cooling block is connected during installation of the device.

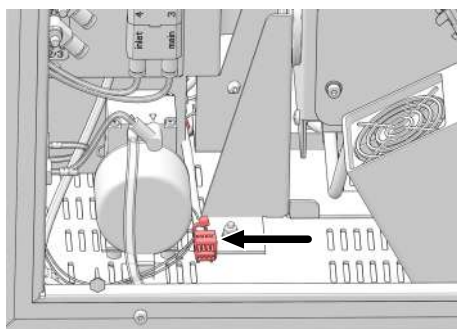


Fig. 7 Electrical connection for Peltier cooling block

3.1.1.2 Combustion system

The combustion furnace is a resistance-heated furnace for digestion temperatures between 700 °C and 1100 °C. Digestion with the combustion tube takes place at temperatures of 1000 °C to 1100 °C.

It is recommended to work at 1050 °C. Higher operating temperatures lead to faster wear of the combustion tube and the furnace.

The samples are placed on quartz boats in the combustion tube. The boats are transported by the ABD.

Combustion tube for standard applications

A reaction reactor (combustion tube) made of quartz glass is used in the digestion system. This combustion tube is used for standard applications.

A flame sensor can be connected to the quartz combustion tube. The sensor monitors the flame and automatically adjusts the speed of the ABD at which the boat is moved into the combustion tube.

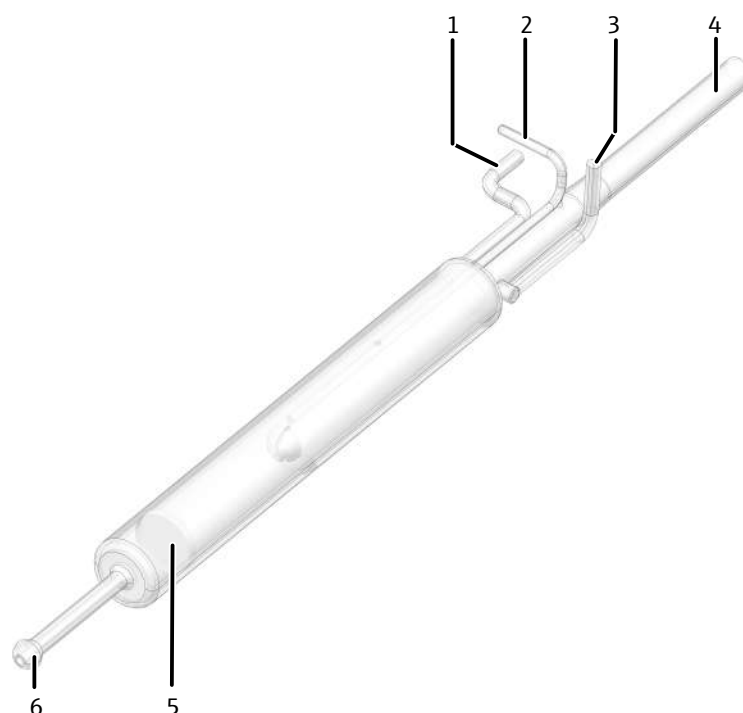


Fig. 8 Quartz combustion tube for standard applications

- | | |
|---------------------------------------|-------------------------------|
| 1 Connection for oxygen supply (main) | 2 Connection for flame sensor |
| 3 Connection for water supply | 4 Input |
| 5 Frit | 6 Outlet with spherical joint |

Combustion tube for high salt loads

A combustion tube with a ceramic inner tube can be used for high salt loads. Ceramics are more resistant to salts than quartz glass.

A flame sensor cannot be used with the ceramic combustion tube, so the ABD transport program must be set or selected in the multWin software.

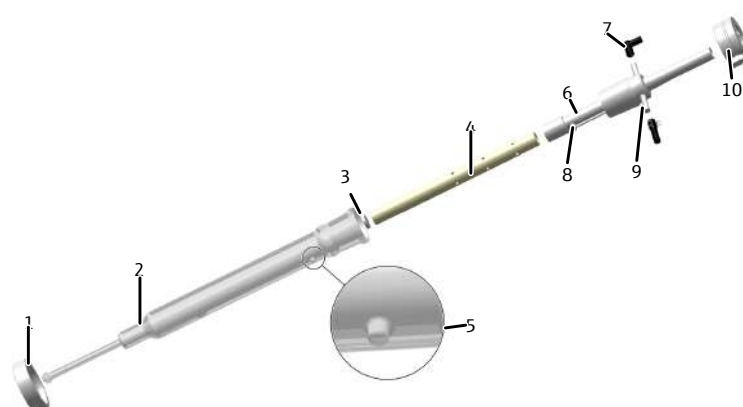


Fig. 9 Ceramic combustion tube for samples with salt loads

- | | |
|------------------------------------|-----------------------------|
| 1 Union nut with screw thread | 2 Outer tube (quartz glass) |
| 3 Frit | 4 Inner tube (ceramic) |
| 5 Nose for positioning | 6 Connection tube |
| 7 FAST connector, angled (oxygen) | 8 Water inlet |
| 9 FAST connector, straight (water) | 10 Clamping screw |

3.1.1.3 Gas supply

The connection between the individual components is made with labeled hoses.

Flow control and gas supply

The argon/oxygen flow as carrier gas is fed to the combustion tube via the ABD. Oxygen is fed into the combustion zone of the combustion tube via a direct connection. The flow rates of the gas flows and the composition of the carrier gas flow are set using the switches on the rear of the dosing modules.

The carrier gas flow can contain only oxygen or consist of a mixture of oxygen and argon.

Connection method

Inside the device, most gas connections have been implemented via FAST connectors (FAST – Fast, Safe, Tight). These connectors provide a tight transition between the hoses and connections with different diameters. The soft sleeves prevent the risk of glass breakage in comparison to rigid screw connections. There are different connector versions.

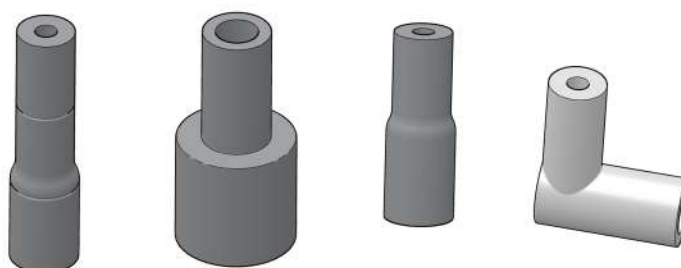


Fig. 10 FAST connector

So-called Fingertight screw connections are also used. These flangeless fittings consist of a conical nipple and a banjo bolt. These hose connections seal purely by tightening the plastic banjo bolt finger-tight.

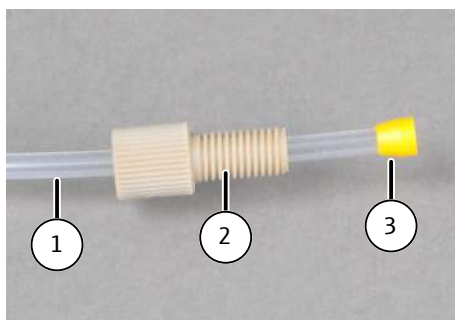


Fig. 11 Fingertight screw connection

- 1 Hose
- 2 Banjo bolt
- 3 Conical nipple

3.1.1.4 Cooling the combustion gases

After thermal digestion, the combustion gases are mixed with absorber solution at the furnace outlet via a T-connector and then passed through a Peltier cooling block. The cooled and completely condensed solution is collected in sample vessels.



Fig. 12 Peltier cooling block with T-connector

- | | |
|--|---------------------------------------|
| 1 Input for absorber solution | 2 T-connector |
| 3 Fork clamp for connection to combustion tube | 4 Peltier cooling block |
| 5 Fastening for block on the heat shield | 6 Connection for sample transfer tube |

3.1.2 Dosing modules

The dosing modules are used to dose water and absorber solution. Dosing is carried out using syringe pumps. The **humidifier module** dosing module doses ultrapure water into the inlet of the combustion tube via a heated transfer line. The **absorber module** dosing module feeds the absorber solution, e.g., ultrapure water or hydrogen peroxide solution, to the combustion gas via the T-connector in the cooling block. During system purging during basic initialization, a small amount of water and absorbent solution is produced as waste, which is collected in a separate waste bottle in the **humidifier module** module.

There are switches on the rear of the modules for setting the flow rates of the process gases and the dosing of water and absorber solution:

- humidifier module – Switch 1 and 2
- absorber module – Switch 3

Switch	Function	Switch position	Meaning
Switch 1	Composition of the process gases	0	Ar + O ₂ main flow O ₂ (at inlet of the combustion tube) + inlet Ar (via the ABD)
		1	only O ₂ main flow O ₂ (at inlet of the combustion tube) + inlet O ₂ (via the ABD)
Switch 2	Flow rate of water/absorber	0	0.2 mL/min 0.2 mL/min of water and absorber solution are dosed respectively.

Switch	Function	Switch po- sition	Meaning
Switch 3	Oxygen flow rate at the inlet of the combus- tion tube (main flow)	1	0.1 mL/min 0.1 mL/min of water and absorber solution are dosed respectively.
		0	200 mL/min
		1	300 mL/min

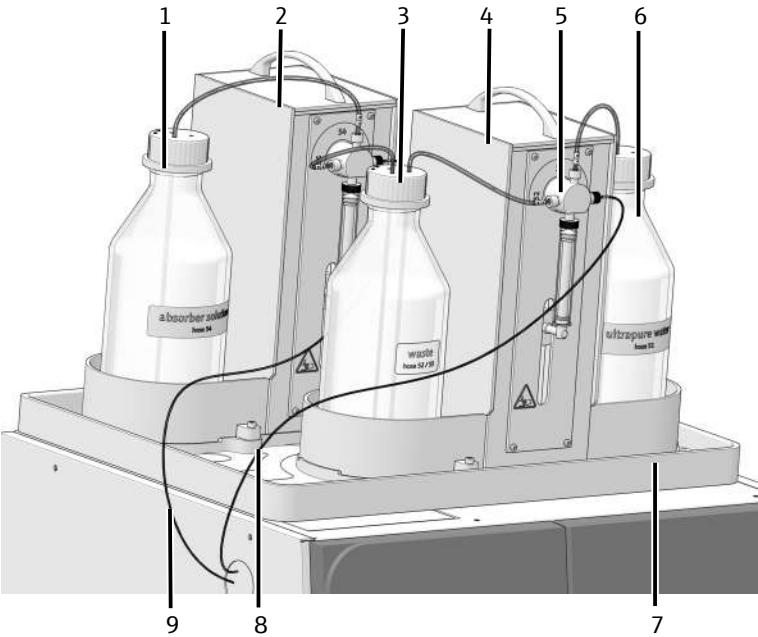


Fig. 13 Dosing modules with storage bottles

- 1 Bottle of absorber solution

3 Waste bottle

5 Syringe pump

7 Drip tray

9 Hose for absorber solution
- 2 Absorber module

4 Humidifier module

6 Bottle for water

8 Hose for water supply

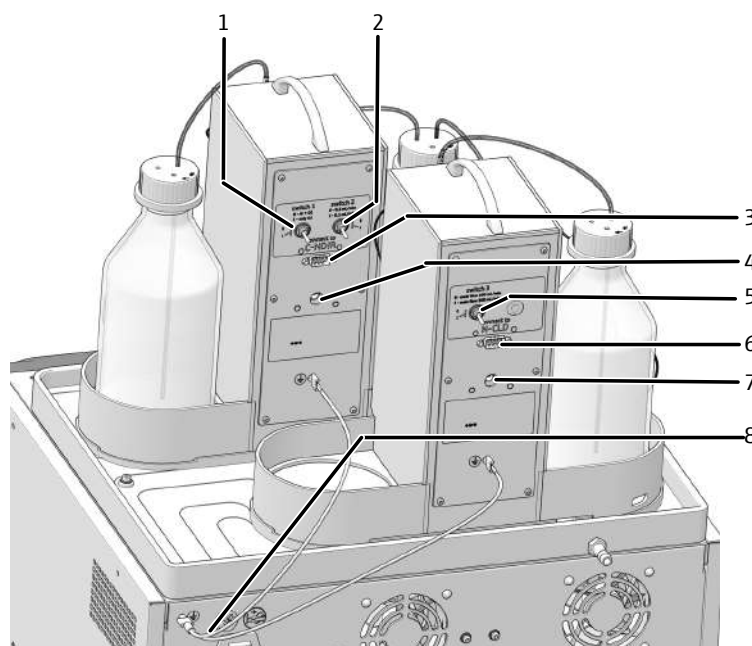


Fig. 14 Connections and switches on the rear of the dosing modules

- | | |
|---|---|
| 1 Switch 1 | 2 Switch 2 |
| 3 Connection to base module (C-NDIR) | 4 External power supply unit connection |
| 5 Switch 3 | 6 Connection to base module (N-CLD) |
| 7 Connection for external power supply unit | 8 Protective conductor for potential equalization |

3.1.3 Automatic Boat Drive (ABD)

The ABD sample feeding module is used to transfer solids and liquids into the combustion tube of the base module.

Solids are weighed into quartz boats and placed in the sample port. Liquids are metered onto the quartz boat through the injection port at the port hatch. The sample is fed either manually or automatically using the MMS.

A quartz glass hook is used to push the loaded quartz boat from the cooled sample port into the combustion tube.

Combustion can be monitored with a flame sensor. This captures the combustion visually and controls the sample boat advance in accordance with the brightness of the flame. Optimizing the combustion characteristics in this way prevents the risk of soot.

Further information on function and design can be found in the separate operating manual for the ABD.

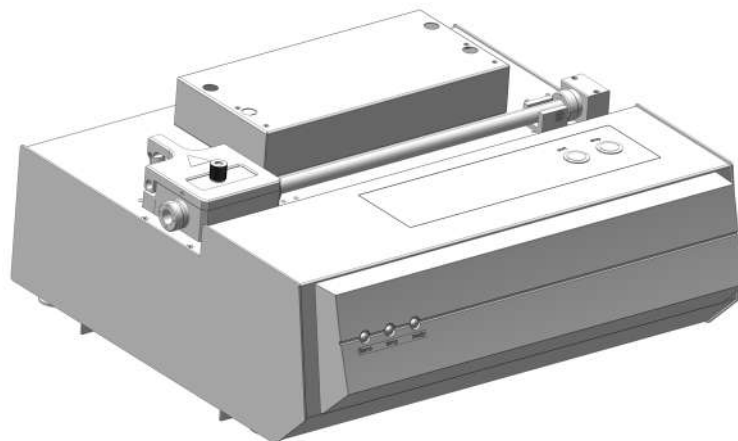


Fig. 15 Automatic Boat Drive (ABD)

3.1.4 Multi Matrix Sampler

The Multi Matrix Sampler - MMS autosampler is a system expansion for the ABD. The MMS automates the feeding of solid and liquid samples. To this end the injector head can optionally be fitted with a dosing syringe to meter the liquids or a gripper to accept the quartz boats. Various sample racks are available for liquids and solids.

The MMS is not included in the scope of delivery of the digestion system, but must be configured and ordered separately.

Further information on installation, operation, and maintenance can be found in the separate operating manual for the MMS.

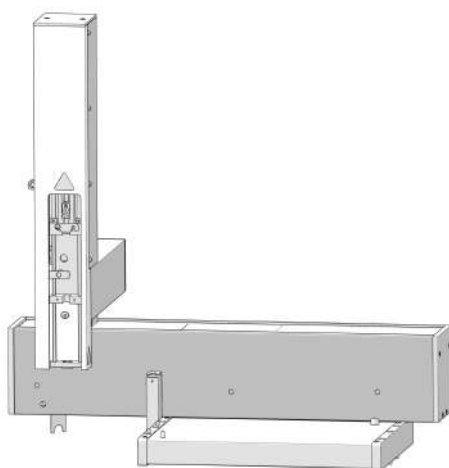


Fig. 16 MMS 5100

3.1.5 Fraction collector for automated operation

The digested samples are collected in the fraction collector during automated sample processing. The fraction collector is based on an AS vario or AS vario ER with a sample tray for 100 sample tubes and a special cannula for dispensing the digested samples into the tubes.

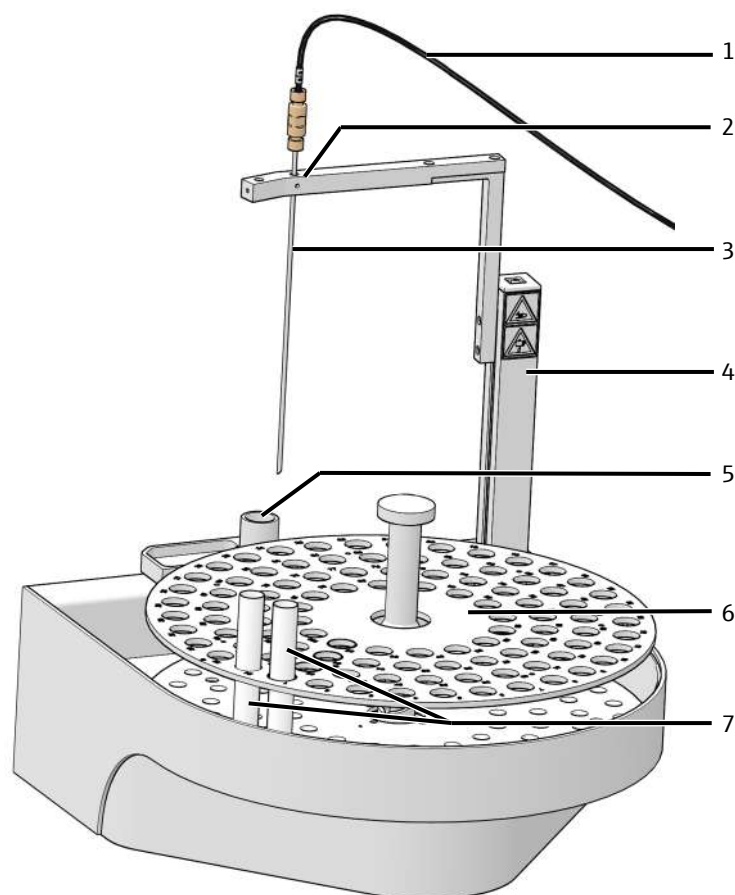


Fig. 17 Structure of the fraction collector

- | | |
|-------------------------------------|-------------------------------|
| 1 Transfer tube for digested sample | 2 Cannula holder |
| 3 Cannula | 4 Autosampler arm |
| 5 Waste container | 6 Sample tray for 100 vessels |
| 7 Sample vessels | |

During automated sample processing with the ICprep digestion system, the fraction collector runs synchronously with the MMS. This means that the first sample on the sample tray of the MMS is transferred to the first position of the fraction collector after digestion, the second sample from the MMS is transferred to the second position of the fraction collector, and so on. The sample trays of the MMS do not have to be filled completely starting with the first position. However, for each occupied sample position on the MMS, an empty sample tube must be placed on the same sample position of the fraction collector.

As part of automated sample processing with the fraction collector, the sample transfer cannula is rinsed internally with absorber solution after each sample. This rinsing solution is dispensed into the waste container of the fraction collector.

The ER fraction collector model can be used as an option. This model is equipped with an additional rinsing vessel in which the cannula is rinsed from the outside with ultra-pure water. This model is particularly suitable if samples with a high halide content are to be analyzed or if additional cannula rinsing is required.

3.1.6 Type plates

The type plates are located on the rear of the device.

The type plate contains the following information:

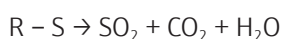
- manufacturer address, trademark
- Designation of the device, serial number
- Electrical connection data
- Conformity markings
- WEEE marking

3.2 Principle of operation

The ICprep digestion system is used for the thermal digestion of solid, liquid, paste, and viscous samples, as well as enriched samples for the determination of sum parameters, such as AOF or EOF. Following digestion, the halide content can be determined using ion chromatography or other suitable detection systems.

Digestion takes place in the horizontally arranged combustion tube by pyrohydrolysis with subsequent thermal oxidation as a one-stage or two-stage process. In the two-stage process, during the first phase of digestion the sample components are pyrolyzed in the argon flow and the generated pyrolysis gases incinerated in the oxygen flow. In addition, small amounts of water in the form of water vapor are automatically injected into the pyrolysis zone to ensure complete conversion to hydrogen halide HX. Next the remaining pyrolysis products are re-incinerated in the oxygen flow during the second phase. In the single-phase process, the entire sample digestion is carried out using only oxygen and the automatic addition of water vapor. The composition of the gases is set at the dosing modules.

The digestion can be summed up by the following equation:



R = carbonic substance

X = F-, Cl-, Br-, I-

The solids and liquids are transferred to the combustion tube with the ABD in quartz boats. The liquids are dosed via the injection port of the sample port into the quartz boats.

After exiting the combustion tube, the reaction gas is mixed with absorber solution, e.g. ultrapure water or hydrogen peroxide solution, in the T-connector and cooled with a Peltier cooling system. The liquid sample digestions can be collected manually or in a fraction collector in sample tubes.

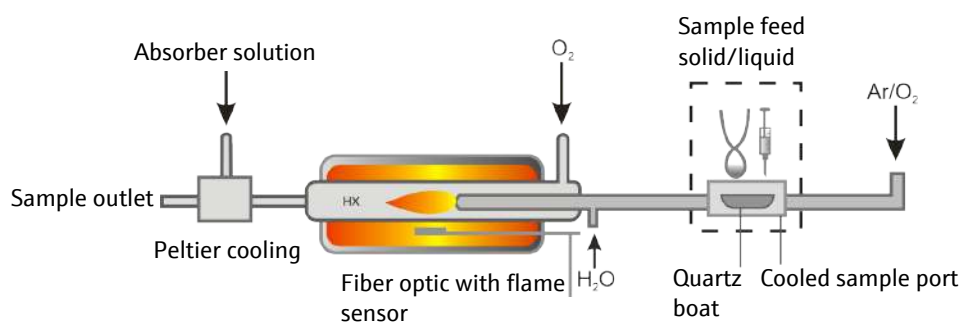


Fig. 18 Functional diagram of ICprep

4 Installation and commissioning

4.1 Installation conditions

4.1.1 Installation location requirements

- This laboratory device is designed for inside use.
- Avoid direct sunlight and radiation from heaters onto the device. If necessary, provide air conditioning.
- The installation site must be free of drafts, dust and caustic fumes. Dust and corrosive vapors can cause damage to the device, e.g., due to corrosion.
- Avoid mechanical shocks and vibrations.
- Do not locate the device near sources of electromagnetic interference.
- Place the device on a heat-resistant and acid-resistant surface.
- Keep the ventilation slits free and do not obstruct them with other devices.

The requirements for the climatic conditions in the operating room can be found in the technical data in this operating manual.

4.1.2 Power supply



WARNING

Danger due to electrical voltage

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

The device operates on single-phase alternating current.

Before connecting the device to a power outlet, check its voltage rating to ensure that the required voltage and frequency match the available power source.

4.1.3 Gas supply

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

The connection hoses are supplied:

- Outer diameter 6 mm
- Inner diameter 4 mm
- length 2 m

4.1.4 Device layout and space requirements

The base module and the other system components are designed as tabletop units. The required space depends on all components that make up the measuring station.

The dosing modules are mounted on the base module.

The ABD is placed to the right of the base module and connected to the combustion tube of the base module.

The ICprep basic model consists of the following components:

- Base module
- Dosing modules
- ABD
- Stand for cannula holder and rack for tubes (not shown)

With the ICprep automatic automation option, the MMS autosampler is also mounted on the ABD and the fraction collector is placed to the left of the base module.

Further information on the workplace:

- There must be a clearance of at least 10 cm between the device system and any cabinet above the system.
- An acid-resistant waste bottle is placed under the bench.
- When using the fraction collector ER: An additional storage bottle of ultrapure water is placed on the bench.

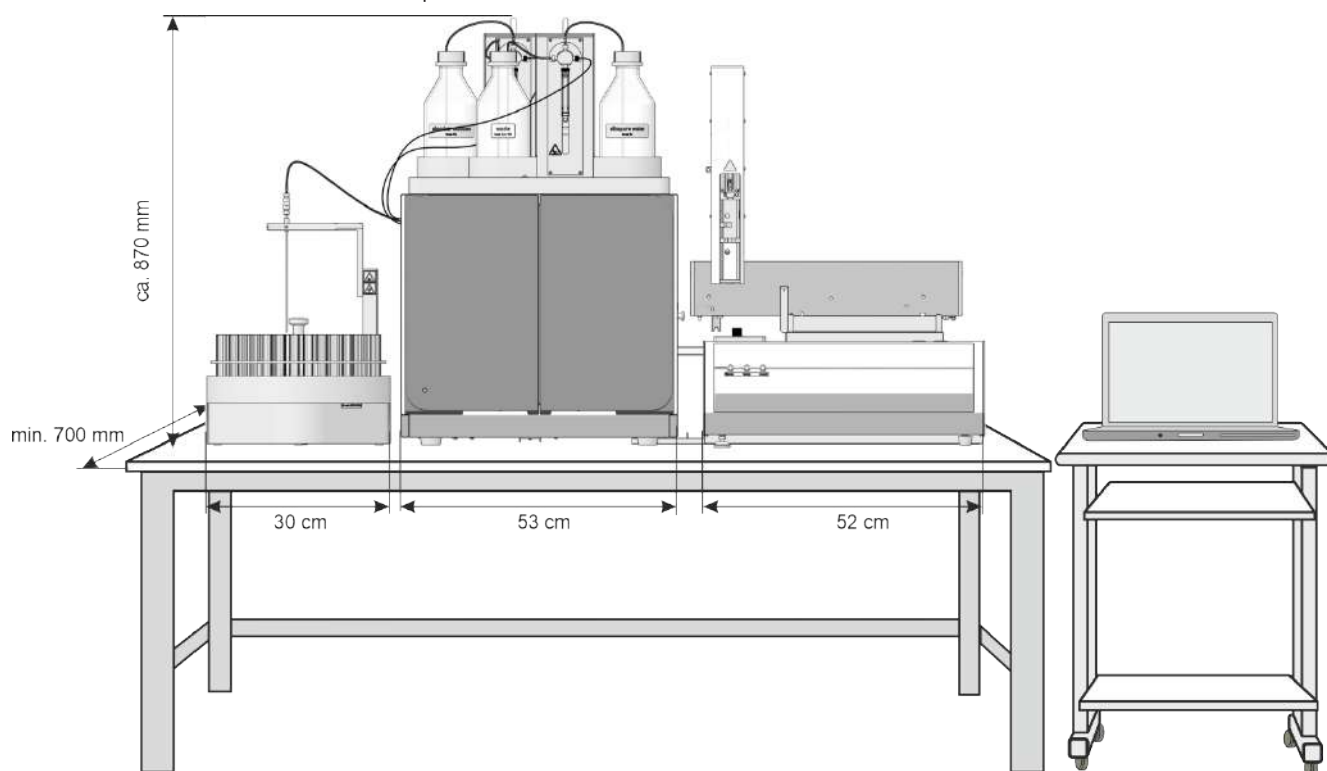


Fig. 19 Space requirements for ICprep automatic digestion system

Component	Dimensions (Width x Depth x Height)	Mass
Base module	530 x 470 x 560 mm	25 kg
Dosing modules	490 x 370 x 320 mm	9.7 kg
ABD	520 x 500 x 210 mm	10 kg
MMS autosampler	400 x 250 x 650 mm	9.5 kg
Fraction collector	350 x 400 x 470 mm	15 kg

4.2 Unpacking and setting up the device

The device will be delivered directly to the final device location by a transportation company. The delivery by this company requires the presence of a person responsible for device installation.

It is imperative that all persons designated to operate the device are present during the briefing given by the service technician.

The device may only be set up, installed and repaired by the customer service department of Analytik Jena or by persons authorized by Analytik Jena.

When installing and commissioning your device, observe the information in the "Safety instructions" section. Compliance with these safety instructions is a requirement for the error-free installation and the proper functioning of your measuring station. Observe all warnings and instructions that are attached to the device itself or displayed by the control and analysis program.

To ensure trouble-free operation, please make sure that the installation conditions are observed.

4.2.1 Installing the digestion system



NOTICE

Connect the electrical connection of the Peltier cooling block by customer service

When installing the digestion system for the first time or when retrofitting an existing EA/AOX analyzer, the AJ service technician must connect the electrical connection for the Peltier cooling block in the base module.

- ▶ Carefully remove the digestion system and its components from the transport packaging.
- ▶ Place the basic module at its intended location. Make sure there is enough space for the other system modules (ABD, autosampler).
- ▶ Connect the dongle to the "external" port on the rear of the base module. Set the switch on the dongle to the "ICprep" position.
- ▶ Connect the electrical connection of the Peltier cooling block. This work may only be carried out by AJ service technicians.
- ▶ Install and connect the combustion furnace (→ "Removing and installing the combustion furnace" 27).
- ▶ Install the dosing modules and the heated transfer line for the ultrapure water supply (→ "Installing dosing modules and transfer line" 28).
- ▶ Install ABD (→ "Installing the ABD" 31).
- ▶ Install the Peltier cooling block (→ "Removing and installing the Peltier cooling block" 31).
- ▶ Install the T-connector (→ "Remove and clean the T-connector" 60).
- ▶ Optionally, install the fraction collector (→ "Installing the fraction collector" 33) and the MMS autosampler (see separate operating manual).
- ▶ Place the waste container under the table. Run the waste hoses from the safety tray and the fraction collector with a continuous downward slope into the waste container. The waste hoses must not be immersed in the liquid. Shorten the hoses if necessary.
- ▶ Connect the base module:
 - Plug in the mains plug to the connection at the rear of the device.
 - Plug the gas hoses into the corresponding "IN O2" and "IN Ar" connections.
 - Connect the PC to the USB port on the base module.
- ▶ Install the combustion tube and connect it to the T-connector. (→ "Installing the combustion tube" 57).

- ▶ Connect the ABD to the combustion tube (→ "Installing the ABD at the combustion tube" 67).
- ▶ Connect the base module to the mains and establish the connection to the gas supply.
- ▶ If the dosing syringes are not yet installed in the dosing modules, install them now (→ "Maintenance of the dosing syringes" 63).
 - ✓ The digestion system is ready for commissioning.

4.2.2 Removing and installing the combustion furnace



WARNING

Danger of electric shock

- Before removing/installing the combustion furnace, switch off the base device via the power switch and disconnect the power plug from the power outlet.



CAUTION

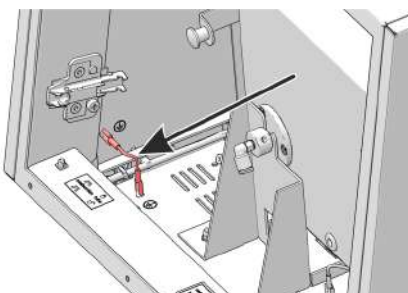
Risk of burns from the hot furnace

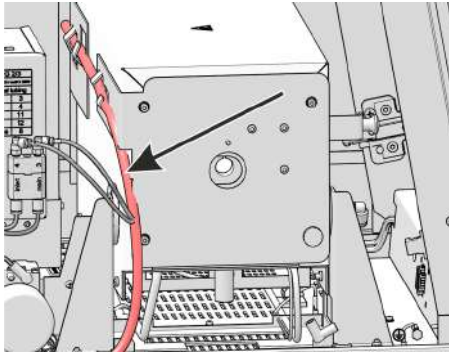
- Allow the device to cool down sufficiently before maintenance.

Removing the combustion furnace

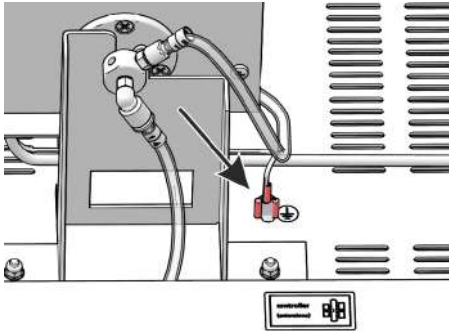
The combustion furnace must be removed for transport.

- ▶ Exit multiWin.
- ▶ Switch off the basic module via the device switch and disconnect the power plug from the socket. Cut the gas supply.
- ▶ Remove the combustion tube (→ "Removing and cleaning the combustion tube" 56).
- ▶ Remove the T-connector (→ "Remove and clean the T-connector" 60) and the Peltier cooling block (→ "Removing and installing the Peltier cooling block" 31).
- ▶ Open or remove the doors from the base module.
- ▶ Remove the left side panel: Disconnect the protective ground conductor. Loosen the screws at the left side panel. Lift the side panel off and store it safely.

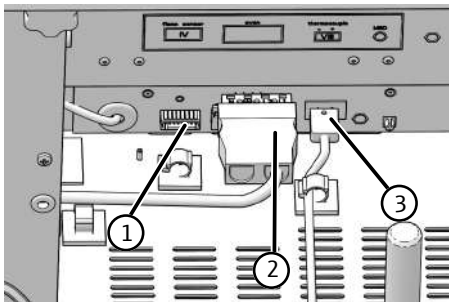




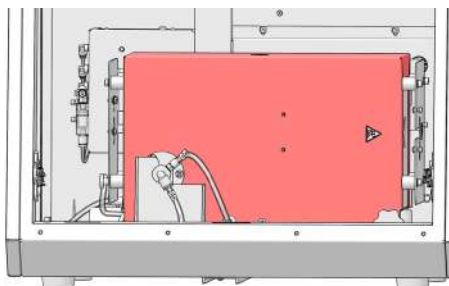
- ▶ Pull the hoses out of the holder on the furnace.



- ▶ Turn the furnace to the vertical position.
- ▶ Disconnect the grounding conductor of the combustion furnace from the floor plate connection.



- ▶ Remove the three plug-in connectors from their sockets:
 - Flame sensor (1)
 - Electrical connection for combustion furnace (2)
 - Thermoelement (3) with orange cable
- ▶ Turn the furnace to a horizontal position.



- ▶ If necessary, loosen hose 14. Press the ring at the plug connector into the connector and pull off the hose.
- ▶ Carefully lift the combustion furnace of the basic module.
 - ✓ The combustion furnace can now be packed.

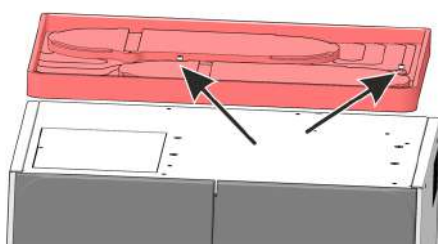
Installing the combustion furnace

The installation of the combustion furnace container is performed in the reverse order to removal.

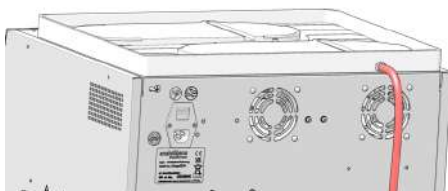
4.2.3 Installing dosing modules and transfer line

The dosing modules are installed on the top of the base module.

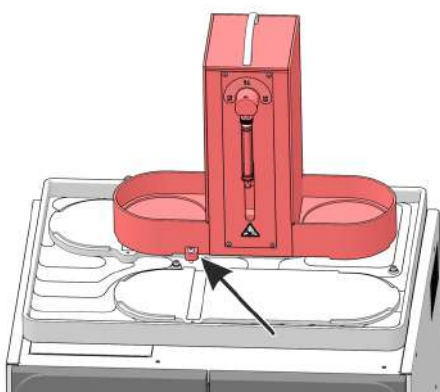
- ▶ Close the opening on the top of the base module with the cover plate.



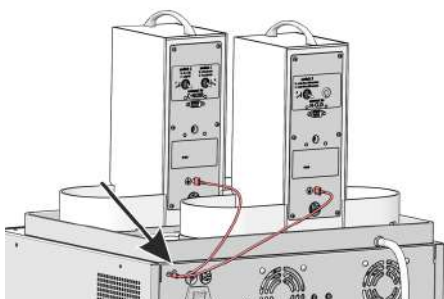
- ▶ Place the safety tray on the top and secure it with the two screws.



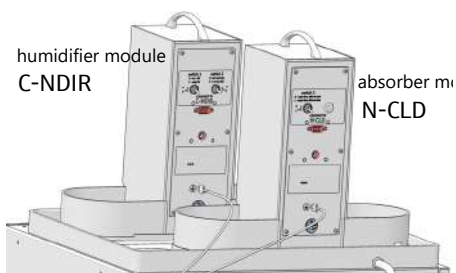
- ▶ Connect the waste hose to the connection on the rear of the safety tray.



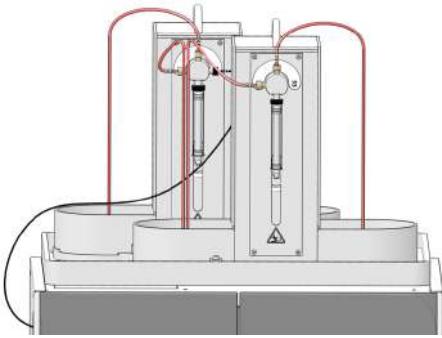
- ▶ Place the dosing modules on the spaces on the safety tray, with humidifier module at the front and absorber module at the rear. Fasten each dosing module with the screw.



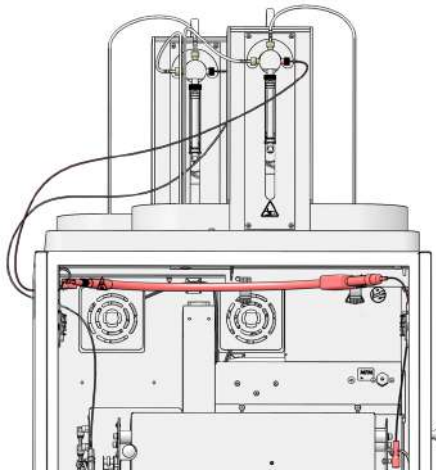
- ▶ Connect the protective conductor of the dosing modules to the connection on the rear of the base module.



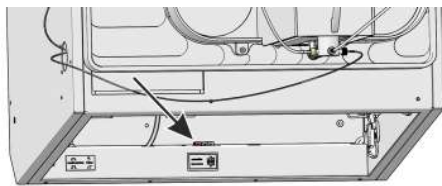
- ▶ Connect the base module to the dosing modules using the interface cables. The interfaces to be assigned to the base module are labeled on the rear of each dosing module: humidifier module at C-NDIR interface, absorber module at N-CLD interface.
- ▶ Plug the external power supply units into the connections on the rear of the dosing modules.



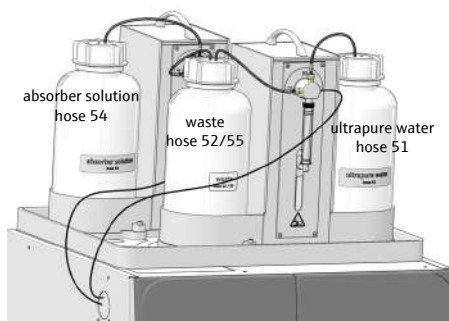
- ▶ Connect the hoses to the dosing pumps. The hoses are numbered. The pumps have labels with the hose numbers to be connected. Do not yet connect the ultrapure water hose (no. 53) to the heated transfer line.
- ▶ Guide the absorber hose (no. 56) to the T-connector through the upper opening in the left side panel of the base module.



- ▶ Attach the heated transfer line to the inside of the top of the base module using hose clamps. The hose end with the FAST connector and the spring clip points to the right. It is later connected to the inlet of the combustion tube.
- ▶ Guide the hose (no. 53) through the opening on the left side of the base module to the outside and attach it to the connection of the syringe pump of the humidifier module.



- ▶ Plug the electrical connection of the heater into the interface on the bottom strip of the front doors.



- ▶ Place the bottle for ultrapure water on the right and the waste bottle on the left in the holders of the **humidifier module**. Place the bottle for the absorber solution on the left in the holder of the **absorber module**. Insert the hoses into the bottles. The bottles are labeled with the hose numbers.
- ✓ The dosing modules are installed on the base module. Once the digestion system is fully installed and ready for commissioning, the dosing syringes must be mounted (→ "Maintenance of the dosing syringes" 63).

4.2.4 Removing and installing the Peltier cooling block

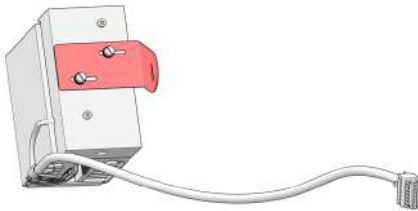


CAUTION

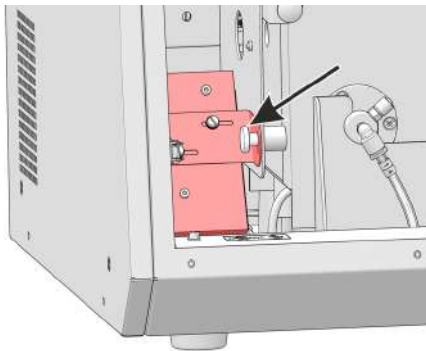
Risk of burns at the hot combustion furnace!

- Only remove and install the cooling block when it is cold.

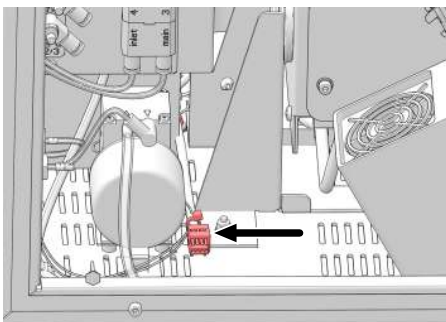
Installing the cooling block



- ▶ Fasten the mounting bracket to the cooling block using two screws. Do not overtighten the screws; the bracket must allow slight movement.
- ▶ Switch off the base module at the mains switch on the rear of the device.
- ▶ Open the front door of the base module.



- ▶ Fasten the cooling block to the bottom right of the combustion furnace using the knurled screws. Tighten the screw only slightly so that the cooling block can still be moved and the T-piece can later be guided precisely to the combustion tube.



- ▶ Connect the plug connector of the cooling block to the cable connection in the base module.
i NOTICE! The cable connection is installed by customer service during device setup.
- ▶ Insert the T-connector (→ "Remove and clean the T-connector" 60).
 ✓ The cooling block with T-connector is installed. Do not connect the T-connector to the combustion tube until the ABD has been installed on the combustion tube.

Removing the cooling block

The cooling block must be removed before transportation.

- ▶ Remove the fork clamp on the spherical joint between the combustion tube and T-connector and separate the connection.
- ▶ The remaining removal steps are carried out in reverse order to installation.
 ✓ The removed cooling block can now be packed.

4.2.5 Installing the ABD



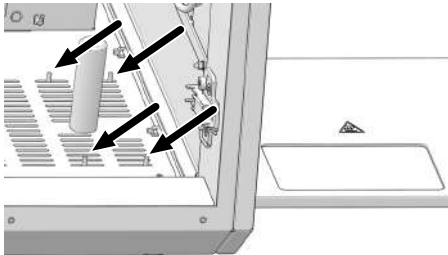
NOTICE

Danger of glass breakage

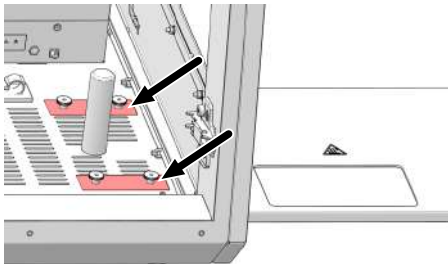
When connecting, the ABD must be aligned to the combustion tube on a guide. Carelessness may result in the quartz glass combustion tube breaking.

First fit the guide for aligning the ABD to the base module and the combustion tube.
Then install the electrical connections and the gas supply.

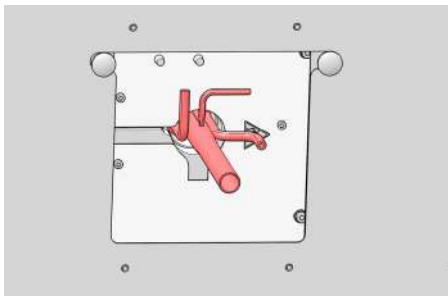
Installing the guide



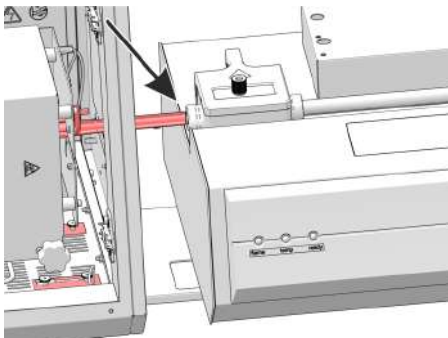
- ▶ Open the front doors of the base module.
- ▶ Turn the combustion furnace to the vertical position.
- ▶ Slide the guide under the right-hand side of the base module so that the fastening pins protrude through the front and rear ventilation slots in the bottom of the base module (see arrows).



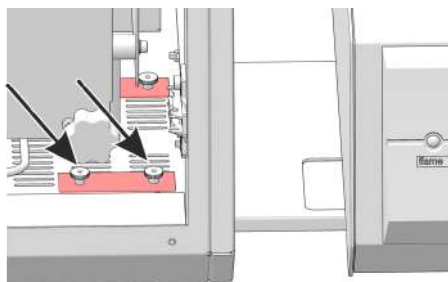
- ▶ Place the perforated plates on the fixing pins of the guide. Screw the plates on loosely. The guide must still be movable.



- ▶ Turn the combustion furnace into the horizontal position and insert the combustion tube into the furnace.



- ▶ Place the ABD on the guide and carefully push it towards the base module.
- ▶ Adjust the height of the ABD using the height-adjustable feet so that the sample port is at the same height as the combustion tube.
- ▶ Align the ABD with the guide so that the combustion tube protrudes into the sample port of the ABD.



- ▶ Tighten the two knurled screws on the front attachment of the guide.

- ▶ Carefully push the ABD back along the guide. Do not move the guide. Pull the combustion tube out of the furnace.
 - ▶ Turn the furnace to the vertical position.
 - ▶ Hand-tighten all knurled screws for fastening the guide.
 - ▶ Check the adjustment: Turn the furnace back to the horizontal position. Insert the combustion tube into the furnace. Push the ABD back onto the base module and check whether the combustion tube can enter the sample port of the ABD without external pressure. If necessary, readjust the height of the ABD and the guide using the steps described above.
- ✓ The ABD guide has been mounted.

Installing electrical connections
and Ar connection

The connections are located on the rear of the ABD.

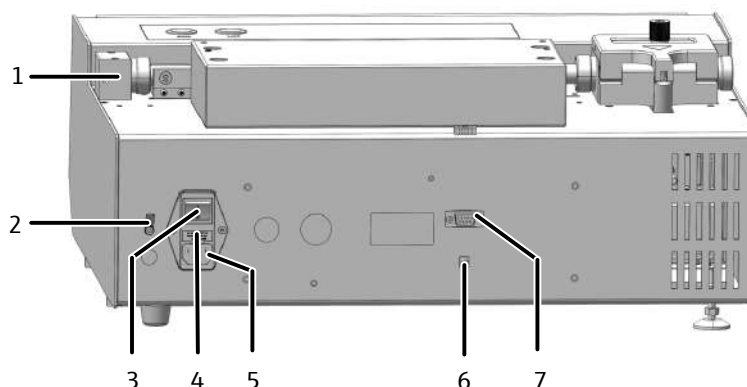


Fig. 20 Connections on the rear of the ABD

- | | |
|---|---------------------------------------|
| 1 Connection hose 16 from gas outlet "Out ABD" on the base module | 2 Earth conductor connection for MMS |
| 3 Power switch | 4 Device fuse |
| 5 Power connection | 6 "Flame" connection for flame sensor |
| 7 Interface to "sampler RS 232" on base module or to the MMS 5100 | |
- ▶ Connect the mains cable to the mains connection on the rear of the ABD.
 - ▶ Connect the flame sensor connecting cable between the ABD and the base module to the "flame" interfaces.
 - ▶ Connect hose 16 for the carrier gas supply to the ABD. Attach the hose with the FAST connector to the "ABD" gas outlet on the base module. Screw the Fingertight connection of hose 16 into the block on the guide tube of the ABD.
 - ▶ Insert the hook into the guide tube (→ "Cleaning and changing hooks" ☞ 69).
- ✓ The ABD can now be installed on the combustion tube.

4.2.6 Installing the fraction collector



CAUTION

Risk of injury from moving parts

There is a risk of injury in the movement range of the sampler arm. For example, hands or fingers may be crushed. The cannula can cause puncture injuries.

- Maintain a safety distance from the device during operation.



NOTICE

Risk of device damage by commissioning with transport locks

If you commission the device with the transport locks still in place, the drives may be damaged.

- Remove the transport locks before commissioning.



NOTICE

Risk of device damage

If the autosampler arm is obstructed during operation, the drives can be destroyed.

- Do not touch the autosampler arm during operation.
- Only carry out manual adjustment when the device is switched off.

Removing the transport lock

The fraction collector is secured for transport with a retaining screw on the bottom of the device. Retain the transport lock for later transport.

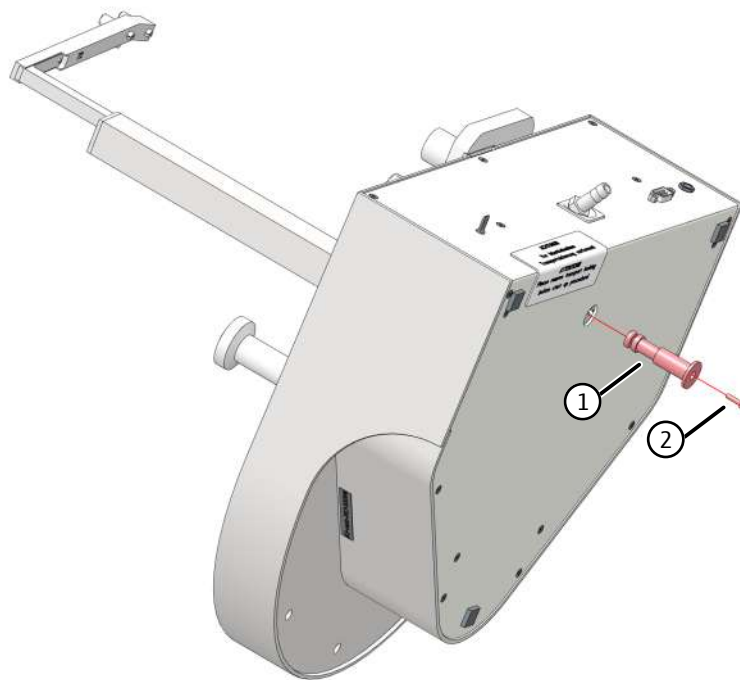


Fig. 21 Transport lock on the fraction collector

- ▶ Turn the device on its side and put it down safely.
- ▶ Remove the screw with the supplied hexagon socket screwdriver. Remove the transport lock (red plastic part).
- ▶ Place the device back on the base plate.

Installing the fraction collector

- ▶ Switch off the base module before installing the fraction collector.
- ▶ Connect the protective conductor to the connection on the rear of the base module. Connect the protective conductor to the connection on the rear of the fraction collector.

- ▶ Connect the cable of the external power supply unit to the connection on the rear of the fraction collector. Do not connect the power supply unit to the mains power supply yet.
- ▶ Connect the fraction collector to the base module using the interface cable (interface on the rear of the fraction collector and "CI-Coul" interface on the rear of the base module).
- ▶ Place the sample tray on the fraction collector. Ensure that it clicks into place.
- ▶ Insert the cannula into the cannula holder.
- ▶ Adjust the height of the cannula manually. In the highest position of the autosampler arm, the tip of the cannula must be 1 to 2 cm above the edge of the vessel and must not touch the vessels when the sample tray rotates.
- ▶ Connect the waste hose to the connection on the waste container. Insert the end of the hose into the waste bottle on the floor.
Lay the waste hose with a continuous slope. Shorten the hose if necessary. The hose must not be immersed in the liquid.
- ▶ Connect the power supply unit to the mains socket.

Install cannula rinsing on the fraction collector ER

- ▶ Place the suitable block with wash cups on the fraction collector.
 - For simpler installation, wet the O-ring on the bottom of the block with water.
 - Fasten the block to the fraction collector with the two hexagon socket screws.
- ▶ Screw the ultrapure water connection into connection (1) and place the hose end in the ultrapure water bottle.
- ▶ Connect the waste hose to the connection on the waste container. Insert the end of the hose into the waste bottle on the floor.
Lay the waste hose with a continuous slope. Shorten the hose if necessary. The hose must not be immersed in the liquid.



Fig. 22 Cannula rinsing on the fraction collector

- | | |
|------------------------------|--------------------|
| 1 Ultrapure water connection | 2 Waste connection |
| 3 Block with wash cups | |

4.2.7 Installing the ceramic combustion tube



CAUTION

Risk of burns from the hot furnace

- Switch off the device and allow it to cool down before replacing components.

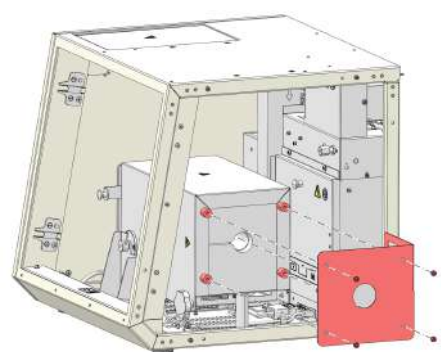


NOTICE

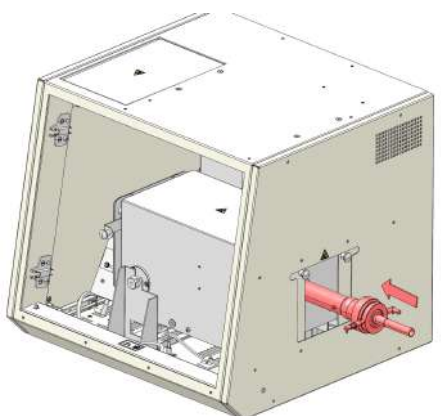
Sweat from your hands can reduce the service life of the combustion tube

Alkali salts in hand perspiration lead to crystallization in the quartz glass when heated.

- Do not touch the combustion tube with your hand. Wear protective gloves.
- Wipe off any finger marks with a cloth wetted with pure alcohol.



- ▶ Exit the multiWin.program. Switch off the digestion system and the gas supply.
- ▶ Remove the quartz combustion tube (→ "Removing and cleaning the combustion tube" 56).
- ▶ Disconnect the protective conductor connection on the right-hand side panel and remove the side panel.
- ▶ Replace the heat shield.
- ▶ Refit the right-hand side panel.



- ▶ Insert the frit (3) into the bottom of the outer tube (2).
- ▶ Connect the inner tube (4) and connection tube (6).
- ▶ Insert the inner tube and connection tube into the outer tube, which is held at an angle. Align the water connection (9) with the lug (5) on the outer tube.
- ▶ Secure the combustion tube with the union nut (1) and clamping screw (10). Tighten the knurled screw slightly.
- ▶ Insert the combustion tube into the furnace. Orient the water connection to the left. This is the only way to insert the reactor into the furnace.

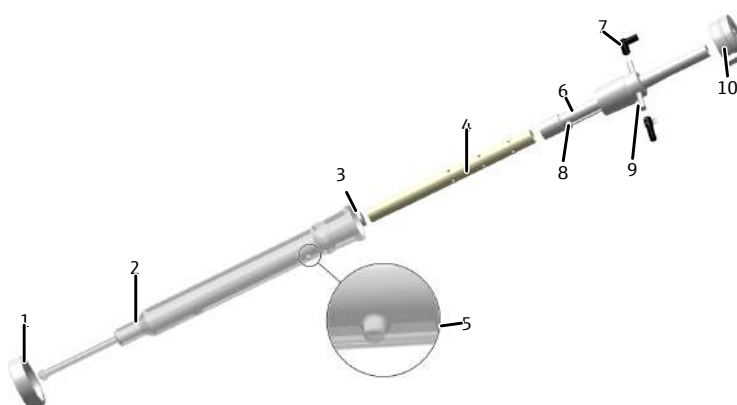
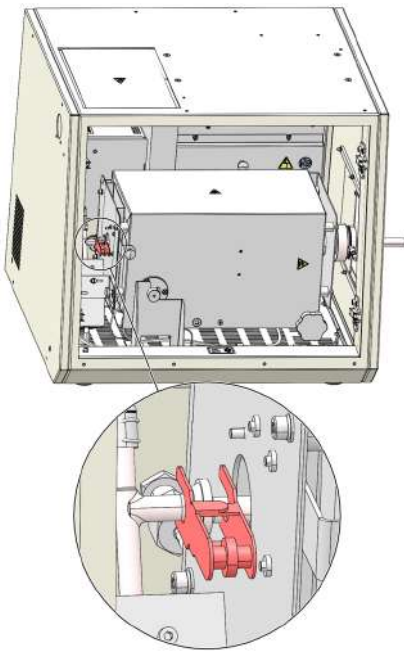


Fig. 23 Install the ceramic combustion tube

- | | |
|------------------------------------|-----------------------------|
| 1 Union nut with screw thread | 2 Outer tube (quartz glass) |
| 3 Frit | 4 Inner tube (ceramic) |
| 5 Nose for positioning | 6 Connection tube |
| 7 FAST connector, angled (oxygen) | 8 Water inlet |
| 9 FAST connector, straight (water) | 10 Clamping screw |



- ▶ Connect the water hose to the combustion tube using the FAST connector. Secure the hose using the clamp.
- ▶ Connect the oxygen hose (no. 3) to the combustion tube. The O₂ connector points to the right.
 ⚠ WARNING! There is a risk of explosion if the hoses are mixed up.
- ▶ Connect the ABD to the combustion tube.
- ▶ Join the spherical joint of the reactor and T-connector. Secure the connection with the fork clamp.

4.3 Converting multi EA 5100 or multi X 2500

The multi EA 5000 (from serial number N7-899/X), multi EA 5100 and multi X 2500 (from serial number N1-399/X) analyzers can be converted into a digestion system using an extension kit. The prerequisite is horizontal operation of the combustion furnace in the base module and the use of an ABD. The upgrade kit contains all the other components required. Version multiWin 1.8.4 is required at a minimum to operate the digestion system.

Converting the analyzer to a digestion system

- ▶ Place the basic module at its intended location. Make sure there is enough space for the other system modules (ABD, autosampler).
- ▶ Connect the dongle to the "external" port on the rear of the base module. Set the switch on the dongle to the "ICprep" position.
- ▶ Remove the combustion tube from the combustion furnace and turn the combustion furnace to the horizontal position.
- ▶ Disconnect the connections on the base module to the detector modules. Set the detector modules to one side.
- ▶ Dismantle the auto-protection valve assembly and remove it from the device (see operating manual for the analyzer).
- ▶ Connect the electrical connection of the Peltier cooling block. This work may only be carried out by AJ service technicians.
- ▶ Install the dosing modules and the heated transfer line for the ultrapure water supply (→ "Installing dosing modules and transfer line" 📖 28).
- ▶ Install ABD (→ "Installing the ABD" 📖 31).
- ▶ Install the Peltier cooling block (→ "Removing and installing the Peltier cooling block" 📖 31).
- ▶ Install the T-connector (→ "Remove and clean the T-connector" 📖 60).

- ▶ When converting to the ICprep automatic version of the device, install the (→ "Installing the fraction collector" 33) fraction collector and the MMS autosampler (see operating manual for MMS).
When converting to the ICprep basic version of the device, an MMS cannot be used.
- ▶ Place the waste container under the table. Run the waste hoses from the safety tray and the fraction collector with a continuous downward slope into the waste container. The waste hoses must not be immersed in the liquid. Shorten the hoses if necessary.
- ▶ Connect the base module:
 - Plug in the mains plug to the connection at the rear of the device.
 - Plug the gas hoses into the corresponding "IN O₂" and "IN Ar" connections.
 - Connect the PC to the USB port on the base module.
- ▶ Install the combustion tube and connect it to the T-connector. (→ "Installing the combustion tube" 57).
- ▶ Connect the ABD to the combustion tube (→ "Installing the ABD at the combustion tube" 67).
- ▶ Connect the base module to the mains and establish the connection to the gas supply.
 - ✓ The digestion system is ready for commissioning.

Converting the digestion system to the analyzer

- ▶ Empty the syringe pumps and hoses of the dosing modules (→ "Maintenance of the dosing syringes" 63). Empty the bottles for ultrapure water and absorber solution, then set them aside.
- ▶ Remove the waste bottle from the dosing module and empty it.
- ▶ Connect the dongle to the "external" port on the rear of the base module. Set the switch on the dongle to the "standard" position.
- ▶ Further conversion to an analyzer is carried out in reverse order to the installation described above. In horizontal operation, the dosing modules can remain on the base module.

4.4 Software installation and device setup

Hardware device setup

The device is set up on the hardware side as a digestion system via the dongle plugged into the rear of the device.

- ▶ Flip the toggle switch down to the **ICprep** position (→ "Electrical components and connections" 13).
 - ✓ The device is now recognized as a digestion system with the dosing modules during device initialization.

Software settings

Install the software according to the "multiWin" operating manual. The software version multiWin 5.8.3 is required at a minimum to operate the digestion system. To be able to use the devices as a digestion system in the software, an external detector must be defined in the device detection.

ICprep

- ▶ When starting the program for the first time, log in as administrator with the user name "Admin" and the password "Admin".
- ▶ Select the **Device | Device - new** menu item.
- ▶ Configure the following settings in the **Device - new** window:

Parameters	Setting
Device type	multi EA 5100
Serial number	Serial number of the device
Elements	external
Combustion tube	CT_HV
States	solid and liquid

Conversion of multi EA 5000 or multi EA 5100

If you are converting an existing analyzer system, you must also configure the device here.

- ▶ Log in to multiWin as an administrator.
- ▶ Select the menu item **Device | Device - edit**.
- ▶ In the **Device - edit** window, configure the above settings for the **Elements**, **States** and **Combustion tube** parameters.

Conversion of multi X 2500

For the AOX analyzer multi X 2500, the **external** option is not available for the **Elements** parameter. To avoid confusion when operating the device as a digestion system, the device should be registered as a multi EA 5100. The device type depends on the device serial number. Replace the first two digits of the serial number of your device with "N7"; the remaining digits of the serial number remain the same.

- ▶ Log in as administrator with the user name "Admin" and the password "Admin".
- ▶ Select the **Device | Device - new** menu item.
- ▶ Configure the following settings in the **Device - new** window:

Parameters	Setting
Device type	multi EA 5100
Serial number	Modified serial number of the device (N7 ...)
Elements	external
Combustion tube	CT_HV
States	solid and liquid

5 Operation

5.1 General information on sample digestion

Observe the following during analysis:

- If you are using the quartz combustion tube, have the sample feed with the ABD preferably controlled automatically by the flame sensor.
- Only use the maximum permissible sample quantities (→ "Technical data for the ICprep" 85).
- Samples and standard solutions with organic solvents can change composition rapidly due to their volatility. Therefore, make sure when preparing and storing samples, that the clear space above the liquid in the sample cup is small. Store the solutions in the fridge.
- Multiple determinations from one sample in automatic mode are not permitted for the "liquid" state. The MMS autosampler runs synchronously with the fraction collector. In a multiple determination, the sample is always taken from the same position. This means that the waste is always discharged into the same position in the fraction collector. As a result, the sample tube overflows onto the fraction collector.

5.2 Switching on the digestion system

Preconditions

Always check the following before switching on the digestion system:

- The other components (ABD, dosing modules, MMS, fraction collector, PC) are connected to the base module and ready for operation.
- The gas supply is connected in accordance with the regulations and the inlet pressure is 6 bar.
- The dosing modules are installed. The hoses are connected correctly. Observe the hose numbers attached to the pumps, supply bottles and hoses.
- The storage bottles for ultrapure water and absorber solution are filled. It is recommended to replace the water and absorber solution every day.
- The waste bottle on the dosing module is empty.
- The waste bottle under the workbench is empty. The waste hoses from the safety tray and from the fraction collector lead into the waste bottle at a continuous downward slope.
- When using the fraction collector ER: The storage bottle for the cannula rinsing solution is filled with ultrapure water.
- The samples and all required solutions are provided.

Switching on

Switch on the digestion module as follows:

- ▶ Open the valves on the pressure reducers of the gas supply.
- ▶ Switch on the system components (ABD, dosing modules, MMS, fraction collector, PC).
- ▶ Switch on the basic module via the power switch.
 - ✓ The basic module boots up. The LED on the left front door illuminates green after approx. 30 s.
- ▶ Check the switch settings for gas flows and the flows of absorber solution and ultrapure water at the dosing modules (→ "Dosing modules" 18).
- ▶ Start the multiWin.program. Log in with user name and password.

- ▶ Click the **[Initialize analyzer]** button.
- ✓ Initialization takes place after successful login.



NOTICE

Observe the run-in period

In the **Status analyzer** window, components that are not yet ready for operation are displayed in red. The heating up time of the combustion furnace to 1050 °C takes approx. 30 min. During the run-in phase it is not possible to start a measurement. The dosing modules and fraction collector are not displayed in the **Status analyzer** window.



NOTICE

Reinitialization required

If the switch settings for the flow settings on the dosing modules have been changed, the device must be re-initialized. This activates the new switch settings.

5.3 Switching off the digestion system



NOTICE

Risk of overheating

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

Switch off the digestion system as follows:

- ▶ Exit multiWin.
- ▶ Switch off the ABD and other system components at the mains switches.
- ▶ Only switch off the base module after a cooling time of 1 hour.
- ▶ Close the gas supply after switching off the modules.
- ▶ Switch off the PC.
- ✓ The digestion system is now switched off.

5.4 Settings in the multiWin software

In the multiWin software, you must create a method and a sequence in preparation for the digestion. The method contains the device parameters, such as furnace temperature and duration of digestion. You can reload a saved method and use it for further digestions.

In the sequence, you save the sample sequence, sample properties and whether the samples are fed into the system manually or by an autosampler. You can also save and reuse sequences.

The functions and settings described here refer to the ICprep digestion system. Detailed information on the functionality of the software can be found in the "multiWin" operating manual.

5.4.1 Create methods

All process parameters required for digestion are stored in a method. At the beginning, you will be guided through the basic settings that are important for the other available parameters. The basic settings cannot be changed later. You then define parameters that can be varied before digestion. The method settings also apply to analyzers with connected detectors (multi EA 5100 or multi X 2500). Measurement evaluations are not provided for in the ICprep digestion system. The corresponding settings are therefore not relevant for digestion. Other parameters are preset for the digestion system and cannot be varied.

- ▶ Select the menu item **Method | Method - new**.
 - ✓ You will be prompted to enter the method name. The method is then saved in the database with this name.
- ▶ Enter the name for the method in the input window and confirm with **[OK]**.
 - ✓ The **Create method** window opens.
- ▶ Configure the basic settings one by one and click on **Next** to confirm.
 - ✓ Once all basic settings have been configured, the **[OK]** button appears.
- ▶ When you click the **[OK]** button, the **Create method** window changes to a window with several tabs where you can define the variable parameters.
- ▶ Define the variable parameters according to the lists below.
- ▶ Click on **OK [authorize]** to save the method settings to the database.
 - ✓ The method can now be used to start a digestion. To change the method, a new version must be created. To do this, use the **Method | Method - edit** menu item.

Settings

Most of the basic settings are preset. You can only select the state of the sample.

Option/Parameter	Description
Name	Display of the method name

Option/Parameter	Description
Parameter	Display external
States	Select solid or liquid
Combustion tube	Display CT_HV
Furnace orientation	Display horizontal
Detectors	Display external Detector
Determination	Can only be set for the solid state Set the value "1". No measurement repetitions are available for sample digestions.

Create method | Global window

The **Global** tab displays the basic settings, which cannot be changed here. You can also find the status of the method here. Methods with the status **authorized** can be used for a digestion. The methods cannot be changed in this status. To edit them, you must create a new version of the method and set the method to the status **in progress**.

Create method | Determination window

Multiple determinations are not permitted for digestion using the ICprep system. The value **1** must always be entered as the number of determinations.

Option/Parameter	Description
Determination	For liquid methods, enter the value "1" here For solid methods, the value is defined in the basic settings and should be "1".
Calculation algorithm	Select any setting The choice of calculation rule has no influence on the digestion.
Operands	Enter presettings for sample volumes or weights The values can be adjusted in the sample sequences.

Option/Parameter	Description
Rinse	For liquid methods only Determine the rinsing cycles for rinsing the syringe with solvent or sample before injection If the Rinse syringe after injection option is enabled, the syringe is rinsed with solvent after the injection.

Create method | Process | System window

Use the **Process | System** tab to specify the parameters for the sample digestion. In the digestion system, the gas flows are partly set via the switches on the dosing modules. By setting switch 1 on the **humidifier module** module, you select the gas flows via the ABD (inlet) and thus the type of digestion. With the setting 0 (Ar+O₂), the digestion takes place in two phases. To start the digestion, argon flows as an inlet gas while the sample is pyrolyzed. When the sample has fully entered the combustion tube, the gas flow is switched to oxygen and post-combustion takes place. During single-phase digestion with the switch set to position 1 (O₂), oxygen flows throughout the entire combustion process.

	Value	Range	Default
Furnace temperature	1050	[0 .. 1,100°C]	1050
2nd combustion	60	[0 .. 900s]	60
Argon (1. phase)	100	[50 .. 200ml/min]	100
Oxygen (2nd combustion)	100	[50 .. 200ml/min]	100
Sample: draw up	20	[10 .. 100* 0.1µL/s]	20
Sample: inject	20	[10 .. 100* 0.1µL/s]	20
▶ minimum cooling time	360	[200 .. 900s]	360

Option/Parameter	Description
ABD	Select the method of transferring the sample to the furnace When using the quartz combustion tube, set the mode to Automatic for operation with a flame sensor. When using the ceramic combustion tube, you must create an individual transport program. It is not possible to connect the flame sensor here.
Furnace temperature	Enter furnace temperature Recommended temperature: 1050 °C
2nd combustion	Enter the duration of post-combustion in the oxygen flow During post-combustion, the argon in the inner tube is replaced by oxygen in order to burn off any pyrolysis residues. Post-combustion begins as soon as the quartz boat has been fully inserted into the combustion tube.
Argon (1. phase)	Argon flow via the ABD (inlet) Two-phase digestion (Ar+O₂) Enter argon flow during pyrolysis (via the ABD) Single-phase digestion (O₂) Entries made here are not taken into account during digestion because no argon flow is present. This gas flow is displayed in the Status analyzer window on the MFC 3. During a single-phase digestion, the display is inactive.

Option/Parameter	Description
Oxygen (2nd combustion)	Oxygen flow via the ABD (inlet) Two-phase digestion (Ar+O₂) Enter oxygen flow during post-combustion Single-phase digestion (O₂) Enter oxygen flow for the entire process This gas flow is displayed in the Status analyzer window on the MFC 2. During pyrolysis in the argon flow, the display is inactive.
Sample: draw up/eject	For liquid methods Enter syringe speed for drawing up and dosing the sample in µL/s
minimum cooling time	For liquid methods Enter the minimum cooling time of the glowing boat after combustion in the ABD port

The flow rate of the oxygen flow directly at the inlet tube of the combustion tube (main flow) is set to 200 or 300 mL/min using the toggle switch on the rear of the **absorber module** module (→ "Dosing modules" 18). A flow rate of 300 mL/min is recommended. This flow remains constant during the entire digestion process.

Create method | Process | Syringe window

For liquid methods

On the **Syringe** tab, you can set the syringe speeds for drawing up and dosing the sample. You can also enter an additional number of strokes and the corresponding speeds to remove bubbles from the sample. For most samples the preconfigured values offer optimum results. Adjustment is recommended for highly viscous or volatile samples.

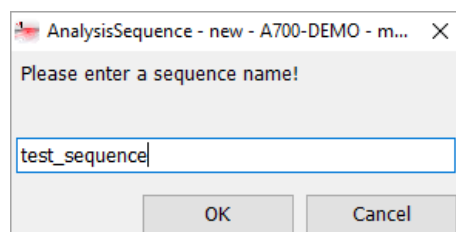
Create method | Detection - External window

Option/Parameter	Description
Detector	External detector
Maximum integration time	Select time for sample digestion

5.4.2 Creating an analysis sequence

In general, an analysis sequence is created before the start of the digestion. However, a sequence can also be prepared, saved and loaded later.

- ▶ Select the menu item **Sequence | AnalysisSequence - new**.



- ▶ Enter the name for the sequence in the input window and confirm with **[OK]**.
 - ✓ The **AnalysisSequence - new** window opens. You will be guided through the settings step by step. Confirm the entries each time by clicking on **Next**.

- Perform the following configuration:

Parameters	Description
Name	Displays the sequence name
Method	Name of the method used for the digestion
Rack Selection	MMS 5000 (...) Automated sample digestion, in which the sample is fed into the ABD port by the MMS manual measurement Manual sample digestion, in which the sample is placed or injected into the sample port of the ABD by hand
First positions	When using the MMS: Set the first occupied position in the autosampler
Number of analysis	Enter the number of samples in the sequence By defining the first position on the autosampler rack and the number of samples, the samples are automatically assigned to the positions of the autosampler in the sequence table. The position and number of the samples can later be modified in the sequence table. Note: During automatic processing, the number of samples is limited to 100 by the available positions on the fraction collector. Sample positions 101 ... 112 on the rack of the MMS autosampler cannot be processed.

- Click [OK].

✓ The **AnalysisSequence - edit** window appears.

- ▶ Click on  to add further lines at the end and click on  to insert them at a selected position in the sequence list.
- ▶ Enter the sample names in the **Name** column. A name must be assigned to each sample, otherwise the sequence line cannot be released for measurement.
- ▶ Optionally enter automatically numbered sample names:
 - Click on .
 - In the input window enter the position of the first sequence item and the number of measurements, the identical part of the analysis name and the initial number.
 - ✓ The samples in the selected sequence lines are automatically assigned the same part of the analysis name and a consecutive, ascending numbering, both separated by an underscore (e.g. "Sample_1").
- ▶ Select the **Sample** sample type in the **Analysis type** column.
- ▶ If necessary, click on a sequence line and enter the initial weight or sample volume for this line on the **Sequence entry** tab in the bottom section of the window. This information has no influence on the digestion.

The Determination number must be 1. A multiple determination cannot be carried out. With liquid methods in automatic operation with synchronously running autosamplers, the digested samples of a multiple determination would be dispensed into the same sample vessel, which would lead to the vessel overflowing.
- ▶ To enable a sequence line, click on . Enable all sequence lines by clicking on .
 - ✓ The released sequence rows have now been prepared for measurement and are highlighted in green.



- ▶ Released sequence rows can no longer be edited. To disable a sequence line again, click on  or to disable all lines, click on .
- ▶ Save all settings by clicking  and close the **AnalysisSequence - edit** window by clicking the **[OK]** button.
 - ✓ The analysis sequence is saved and can be loaded for a digestion.

5.4.3 Create a transport program for the ABD

In multiWin, specific data sets tailored to samples and methods can be saved in addition to the preset transport parameters of the ABD and linked to the method (**Process** tab).

Use the flame sensor for digestion in the quartz combustion tube. Select the **Automatic** mode as the transport mode in the method. The combustion is controlled fully automatically via the evaluation of the flame sensor. The mode is universally suitable for liquid and solid samples, changing sample matrices and sample quantities.

The flame sensor cannot be used for digestion in the ceramic combustion tube. In this case, you must define a transport program for the transfer into the combustion tube with hold points, wait times and feed speeds in advance. In the method, select the **Parameter** mode for the ABD and a previously created parameter set.

Editing ABD parameters

Create the parameters of a transport program for the ABD as follows:

- ▶ Select the menu item **Method | ABD Parameter - edit**.

✓ The **ABD** window appears.

- ▶ Click on  to create a new program.
- ▶ Enter a name for the new program in the input window.
- ▶ The **ABD** program window appears.
- ▶ Define the following parameters:

Option	Description
Name	Name of the parameter set The name can be edited after clicking on Edit .
Mode	Select the Parameter option
Status	Edit status of the ABD program
State	Select the state of the sample
Description	Optionally enter a short description
Remark	Optionally enter text for using the transport parameters Note: The transport parameters list can also be filtered by the Description and Remark options.
Parameter	Enter the following parameters for each program step: Speed: Feed rate Position: Hold point Waiting period: Wait time at the hold point You can define up to three program steps. Click  to add another step. Click  to remove a selected line.

- ▶ Select the **authorized** option from the **Status** list.
 - ▶ Click **[OK]** to confirm all entries.
- ✓ The ABD program can now be used in a method.

You can copy an existing ABD program by clicking on  and then modify it. Highlight data records that are no longer needed on the left-hand side of the **ABD** window and then click on  in the toolbar of the ABD window. ABD programs that are used in a method cannot be deleted.

5.5 Start digestion

Preconditions

- The digestion system is switched on and initialized.
- The switches of the dosing modules for setting the process gases and the flows of ultrapure water and absorber solution are in the desired position (→ "Dosing modules"  18).
- The system has been checked for leaks (→ "Checking the system for leaks"  71).
- A method for digestion has been created and activated (→ "Create methods"  43).

Start sample digestion

i NOTICE! The switch settings of the (→ "Dosing modules"  18) dosing modules become active when the device is initialized. Changing the switch position requires the device to be re-initialized.

- ▶ During device initialization, the last method used is activated and the switch positions of the dosing modules are read out. Another method can be activated in the main window by clicking on the **Method - activate** menu command.
- ▶ Click on **[Start Measurement]** in the main window.
 - ✓ The **AnalysisGroup - Select** window appears.
- ▶ To save the sample data, select an analysis group and close the window by clicking on **[OK]**.

Alternatively, click on  to create a new analysis group and enter a name.

- ▶ In the **AnalysisSequence - Select** window, select an existing analysis sequence and edit it if necessary.

Alternatively, click on  to create a new sequence.

- ▶ After activating the sequence entries (→ see above), click on **[OK]**.
- ▶ In the **Measurement - Analysis** window, click on **[Start Measurement]**.
 - ✓ The digestion sequence starts and the process data is displayed on the screen.

5.6 Sample feed for the digestion system

5.6.1 Prepare the quartz boats for the sample feed

Samples are transferred with the ABD on quartz boats into the combustion tube for digestion.

Liquids and solids

The quartz boat is covered with quartz fleece as a carrier material for measurements of liquids and solids in order to prevent rapid, uncontrolled evaporation/spraying of the samples and thus system contamination or incomplete combustion. The quartz fleece also acts as sacrificial material and binds any ionic contamination contained in the samples (e.g. alkaline and alkaline earth metal ions) and can thus contribute to an increased service life of the quartz glass components.

- ▶ Use clean scissors to cut a sufficiently large piece from the quartz fleece supplied. The piece must cover the floor of the boat and must not protrude at the sides. Only use a single undamaged piece of quartz fleece as carrier material! When using several small or damaged pieces, correct functionality cannot be ensured.
- ▶ Place the quartz fleece on the bottom of the quartz boat.
- ▶ To prevent blank value problems due to contamination, treat the quartz boat and quartz fleece as follows:

- Bake the quartz boat and quartz fleece before using them for the first time.
- Only handle baked quartz boats/fleece with gloves.
- Store quartz boats/fleece in a desiccator or screw-top bottle to prevent contamination.



Fig. 24 Quartz boat with correctly inserted quartz fleece

Quartz container with enriched samples

No quartz fleece is required for digestion using quartz containers on which samples are enriched on activated carbon for the determination of sum parameters.

- ▶ Bake the quartz boats before using them for the first time. Store and treat baked quartz boats as described above.
- ▶ Place the quartz container in the boat without quartz fleece.



Fig. 25 Boat with quartz container

Filter with enriched samples

Filters with samples enriched by the batch method are digested in quartz boats with retainers.

- ▶ Bake the quartz boats before using them for the first time. Store and treat baked quartz boats as described above.
- ▶ Slide the filter under the retainer in the quartz boat.



Fig. 26 Boat with retainer for filters

5.6.2 Sample feed in automatic operating mode



CAUTION

Crushing risk when closing the sample port!

High forces occur when closing the sample port of the ABD.

- Do not reach into the port area when closing.



CAUTION

Risk of injury to the hand in the movement area of the autosampler

High forces are generated when the autosampler arm is moved. If you are not careful, you could crush your hand or perforate it on the needle.

- Do not reach into the movement area of the autosampler during operation.
- Do not place any samples on the sample trays during operation.



NOTICE

Solvent vessel collides with port cover

The solvent vessel of the MMS autosampler is located in the opening area of the port cover and may collide with the port cover.

- When using the liquid rack, remove the solvent vessel before opening the port.
- Do not place the solvent vessel on the MMS when the solid rack is installed.

Loading the autosampler and fraction collector

In automatic mode, samples are fed to the ABD using the MMS autosampler. The digested samples are collected in the fraction collector in sample tubes provided. No sample positions can be defined for the fraction collector in the sequence in multiWin. It therefore runs synchronously with the MMS autosampler. A sample from an MMS sample position is placed in the same position on the fraction collector after digestion. Therefore, for each occupied sample position on the MMS, place an empty sample tube on the sample tray of the fraction collector. A maximum of 100 samples can be processed in one sequence. The number results from the sample positions of the fraction collector. Positions 101 ... 112 on the rack of the MMS cannot be processed.

For information on adjusting, maintaining, and preparing samples on the MMS, refer to the MMS operating manual.

Preconditions

- The MMS autosampler is mounted on the ABD and calibrated.
- The fraction collector is located on the left-hand side of the base module and connected to the system.
- The cannula rinsing system is installed for the fraction collector ER and the ultrapure water bottle is positioned next to the device.

Solid samples

- ▶ Check that there are no quartz boats in the sample port.
- ▶ Weigh the solid sample into the prepared quartz boats or place the quartz containers with activated carbon on the quartz boats.
- ▶ Place the quartz boats on the solid rack of the autosampler.
- ▶ Place the sample tubes on the fraction collector according to which positions are occupied on the autosampler.
- ▶ Start the digestion in multiWin.

Liquid samples

- ▶ If used, remove the solvent vessel from the MMS autosampler. It could collide with the port cover.
- ▶ Open the sample port by pressing the "port" button on the ABD.
- ▶ Use tweezers to insert a prepared quartz boat with quartz fleece into the open sample port. The eye of the boat must sit on the quartz hook.
- ▶ Close the sample port by pressing the "port" button again.
- ▶ Reattach the solvent vessel to the autosampler.

- ▶ Place the sample vessels with the liquid samples on the liquid rack of the autosampler.
- ▶ Place the sample tubes on the fraction collector according to which positions are occupied on the autosampler.
- ▶ Start the digestion in multiWin.

5.6.3 Sample feed in manual operating mode

In manual operating mode, each sample is manually loaded into the ABD. The sample digestions start individually after a prompt from the software. The digested sample is transferred via the sample transfer tube with cannula on the T-connector into a sample tube provided. The scope of delivery includes a stand with a clamp for fixing the cannula and a rack for holding the sample tubes. Fix the cannula to the stand so that the tip of the cannula is almost touching the bottom of the sample tube, but does not hit the bottom of the tube.



Fig. 27 Attach the cannula to the stand



CAUTION

Crushing risk when closing the sample port!

High forces occur when closing the sample port of the ABD.

- Do not reach into the port area when closing.

Solid sample

- ▶ Weigh the solid samples into the boat prepared with quartz fleece. Place quartz container or filter in quartz boat (→ "Prepare the quartz boats for the sample feed" 50).
- ▶ Start the digestion (→ "Start digestion" 50).
- ▶ When prompted by multiWin, insert the quartz boat into the open sample port using tweezers. Place the eye of the boat on the hook.
- ▶ Click on **OK** to confirm that the quartz boat has been inserted.
 - ✓ The sample port closes. After a rinsing time that can be configured in the method, the transport of the quartz boat into the combustion tube begins.

Liquid sample

- ▶ Bake a quartz boat prepared with a carrier material (→ "Prepare the quartz boats for the sample feed" 50).
- ▶ **i** NOTICE! Possible system contamination! If a quartz fleece is not used, the entire sample may evaporate or splash in an uncontrolled manner. This means that the sample is not digested and the system can become contaminated.
- ▶ Open the sample port using the "port" button.
- ▶ Use tweezers to insert the quartz boat into the open sample port. Place the eye of the boat on the hook.
- ▶ Close the sample port using the "port" button.
- ▶ When prompted by multiWin, dose the liquid sample directly onto the carrier material of the boat using a microliter syringe through the septum of the injection port.
- ▶ Click on **OK** to confirm dosing.
- ▶ Start the measurement.
 - ✓ The quartz boat is transported into the combustion tube.

6 Maintenance and care

6.1 Maintenance intervals

ICprep

Maintenance interval	Maintenance task
Daily	Check gas flow
Weekly	Clean and check device and system components
Weekly	Check all screw connections for tight fit
Monthly	Check fastening screws for tight fit
As required, if gas path blocked	Replace particle filter in the gas inlet
As required, if gas path blocked	Replace non-return valve at the gas
As required	Replacing the cannula

Combustion tube and T-connector

Maintenance interval	Maintenance task
Monthly	Check for cracks and damage
Monthly	Check FAST connector for tight fit, cracks or damage
As required, in case of devitrification and cracks	Replace the combustion tube and T-connector
As required	Clean combustion tube and T-connector

Dosing modules

Maintenance interval	Maintenance task
As required, in case of damage or soiling	Maintain or replace dosing syringes
As required	Replacing tubes
As required	Clean and replace storage bottles and waste bottles

ABD

Maintenance interval	Maintenance task
Daily	Check the injection port with septum for leaks
Weekly	Cleaning the device
Weekly	Check hose connection (hose 16) for tight fit and leaks
Weekly	Check seals
As required	Clean port
As required, if leaking	Replace septum in injection port
As required, in case of devitrification of the surface	Replace quartz boat
As required, in case of discoloration	Replace carrier material in the quartz boat
As required	Clean the hook and guide tube
As required	Replace the seal in the sample port

MMS

Maintenance interval	Maintenance task
Weekly	Cleaning the device
As required	Replace the septum on the solvent vessel
As required	Replace quartz boat
As required, in case of discoloration, hardening or increased brittleness	Replace carrier material in the quartz boat
As required	Cleaning the cannula guide
As required	Replacing the cannula

Information on maintenance and calibration of the autosampler can be found in the "Multi Matrix Sampler" operating manual.

Fraction collector

Maintenance interval	Maintenance task
Weekly	Cleaning the device

6.2 Cleaning the device

- Wipe up spilled samples in the base module or on accessories immediately with blotting paper.
- Use a soft, clean cloth to wipe away any contamination from the device. A commercially available neutral cleaning agent can be used for maintenance of the casing.
- **i** NOTICE! Alcohol, organic solvents, and abrasive cleaners can damage the paint.
- Do not use dripping cloths. Do not allow liquids to enter into the device.

6.3 Maintenance of the base module

6.3.1 Removing and cleaning the combustion tube



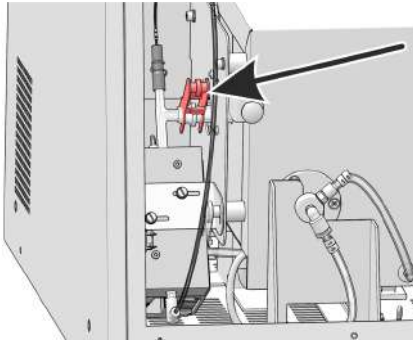
CAUTION

Risk of burns on the hot furnace and combustion tube

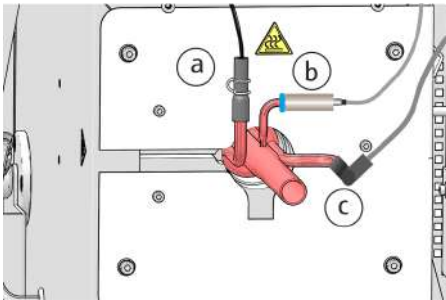
- Allow the device to cool down after digestion. Only remove the combustion tube when it has cooled down.
- Wear the heat-proof gloves included in the delivery when handling hot components. These gloves are suitable for temperatures of up to 200 °C.

Removing the combustion tube

- ▶ Exit the multiWin program.
- ▶ Switch off the device system at the mains switches. Pull the plug out of the mains sockets.
- ▶ Switch off the gas supply.
- ▶ Open the front doors of the ICprep.

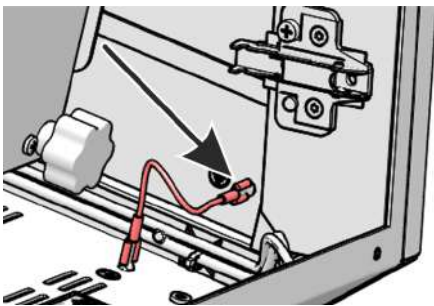


- ▶ Disconnect the fork clamp from the spherical joint between the T-connector and the combustion tube on the left-hand side of the device.
- ▶ Disconnect the ABD from the combustion tube and push it to the side (→ "Disconnecting the ABD from the combustion tube" 66).



- ▶ Carefully disconnect the water hose (a), the flame sensor (b) and hose no. 3 (c) from the combustion tube connections.
- i** NOTICE! Risk of glass fracture! The glass connection of the flame sensor can break very easily. Hold the gas connection with one hand whilst gripping the blue ring at the sensor with the other hand and pull off the sensor. Do not twist the sensor!
- ▶ Carefully pull the combustion tube out of the combustion furnace to the side.

✓ You can now clean the combustion tube.



If the connections via the sliding doors in the right-hand side panel are difficult to reach, you can remove the right-hand side panel:

- ▶ Disconnect the protective conductor connection on the right-hand side panel.
- ▶ Loosen the four fastening screws on the side panel and lift out the panel.

Cleaning the combustion tube

- ▶ Check the combustion tube for excessive crystallization, cracks and blow-outs.
- ▶ If necessary, clean the combustion tube using a suitable solvent and brush or cellulose.
- ▶ Alternatively, burn out the combustion tube for cleaning in a muffle furnace or with a suitable burner flame.
- ✓ You can now reinstall the cleaned combustion tube. Only use intact combustion tubes.

6.3.2 Installing the combustion tube



CAUTION

Risk of burns at the hot combustion furnace!

- Only install the combustion tube when the device is cold.



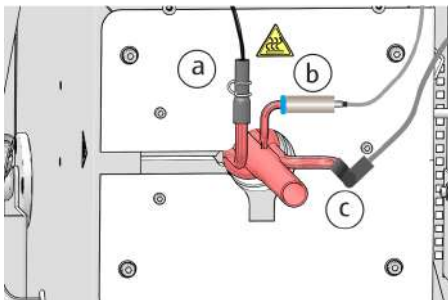
NOTICE

Damage to the combustion tube

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

- Do not touch the combustion tube with your bare hands. Wear gloves.
- Before inserting, wipe the outside of the combustion tube with ethanol and cellulose.

Installing the combustion tube

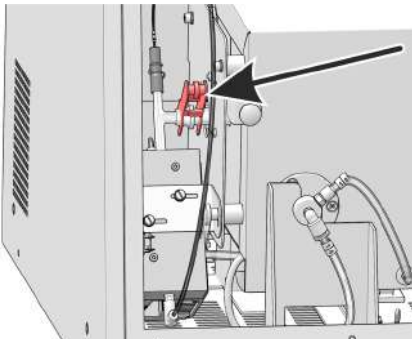


i NOTICE! Possible damage to the device! Do not use damaged combustion tubes with cracks or devitrification.

- ▶ Insert the combustion tube into the combustion furnace.
- ▶ Connect the FAST connector of the water hose to the combustion tube (a).
- ▶ Connect hose no. 3 for the oxygen supply to the combustion tube (c) using the angled FAST connector.

⚠ WARNING! Risk of explosion or carbonization! The hoses at the combustion tube must not be swapped!

- ▶ Slide the flame sensor (b) onto the connection on the combustion tube. Hold the glass connection with one hand and push the sensor onto it with the other. Do not twist the sensor!



- ▶ Connect the ABD to the combustion tube (→ "Installing the ABD at the combustion tube" 67).
 - ▶ Open the doors of the digestion system.
 - ▶ On the left-hand side of the device, join the spherical joint of the combustion tube and T-connector and secure the connection with the fork clamp.
- ✓ The combustion tube is now installed and connected.

6.3.3 Replacing the hose connections

FAST connectors connect hoses with glass components. Use the threading aid to feed thin hoses into the connectors. This is included with the analyzer. Check the system for leaks after hose replacement.



- ▶ Slide the FAST connector onto the canula of the threading aid. The narrow hole of the connector points upwards.



- ▶ Thread the hose into the canula of the threading aid.



- ▶ Slide the FAST connector from the canula onto the hose.
- ▶ Pull the hose out of the canula of the threading aid. Pull the hose of the FAST connector until it no longer reaches into the wider hole.

Angled FAST connectors

With angled FAST connectors, do not slide the hose ends beyond the side length of the connector. The gas flow will otherwise be impaired.

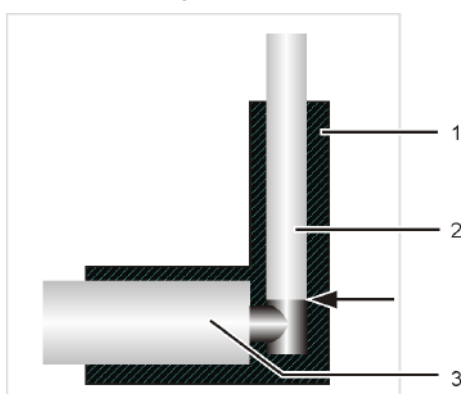


Fig. 28 FAST connector, angled

- 1 Angled FAST connector
- 2 Hose
- 3 Glass connection

Fingertight connections

- ▶ When replacing Fingertight connections, only use straight cut, round, uncrimped hose ends.
- ▶ Slide the conical nipple onto the hose with the conical side towards the banjo bolt. The conical nipple and hose end must be flush.
- ▶ Do not jam the banjo bolt during insertion and only tighten it hand-tight.

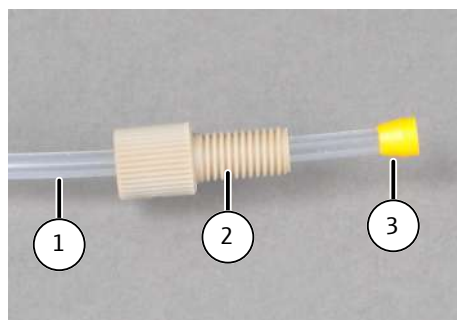


Fig. 29 Replacing the Fingertight connection

- | | |
|------------------|--------------|
| 1 Hose | 2 Banjo bolt |
| 3 Conical nipple | |

6.3.4 Remove and clean the T-connector

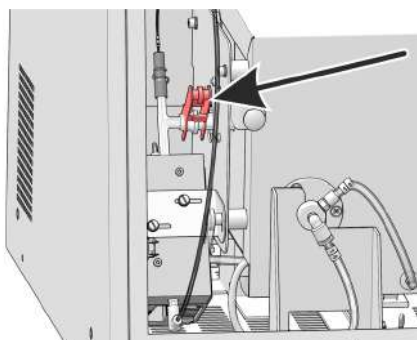


CAUTION

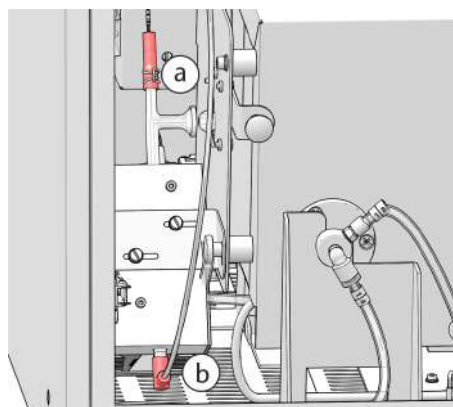
Risk of burns from the hot combustion furnace

- Only remove and install the T-connector when cold.

Removing the T-connector



- ▶ Switch off the digestion system at the device switch and open the front doors.
- ▶ Remove the fork clamp on the ground joint to the combustion tube.



- ▶ Remove the absorber hose (no. 56) from the upper end of the T-connector. To do this, use pliers to push the spring wire clamp on the FAST connector upwards. Carefully pull the FAST connector off the T-connector (a).
- ▶ Pull off the sample transfer tube (no. 50) with the angled FAST connector from the lower end of the T-connector (b).
- ▶ Pull the T-connector out of the cooling block.

Cleaning the T-connector

- ▶ Check the T-connector for deposits and cracks.
- ▶ Rinse the T-connector with ultrapure water.

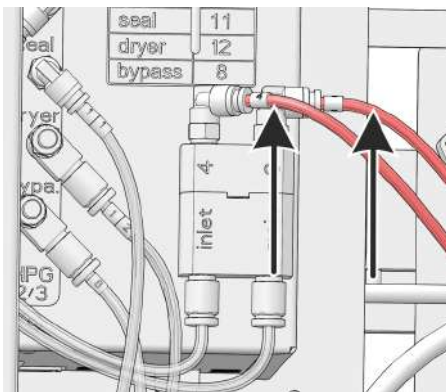
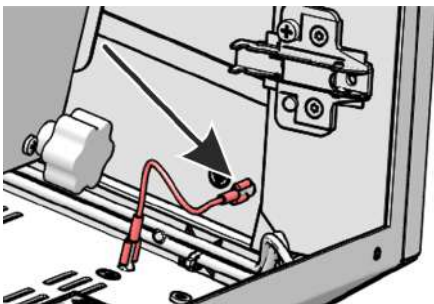
Installing the T-connector

Installation is carried out in reverse order to removal above.

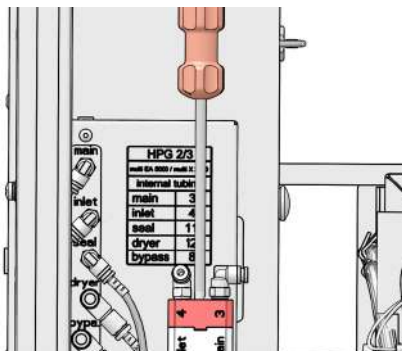
6.3.5 Replacing the check valves on the gas box

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

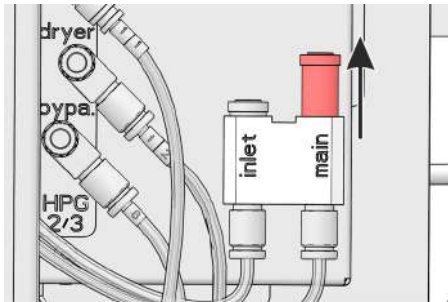
- ▶ Switch off all devices in the system at the mains switches.
- ▶ Cut the gas supply at the shut-off valve.
- ▶ If the device needs to be turned to reach the left-hand side: Disconnect the ABD from the combustion tube and carefully push the ABD to the side (→ "Disconnecting the ABD from the combustion tube" 66). Disconnect the connections between the ABD and the base module.
- ▶ Disconnect the H₂O hose and the absorber hose from the connections on the pump modules. Collect leaking liquids with cellulose.
- ▶ Unscrew the transfer tube from the cannula of the fraction collector.
- ▶ Pull the hoses through the openings in the side panel into the inside of the device.
- ▶ Remove the protective ground conductor on the left side panel. Loosen the 4 screws on the left side panel and lift off the side panel.



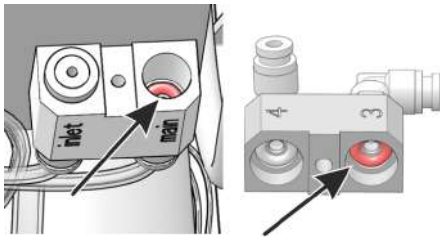
- ▶ Pull hoses 3 and 4 out of the connection on the valve block (see arrows).



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



- ▶ Remove the upper part of the valve block and take the check valve out of the "main" connection.

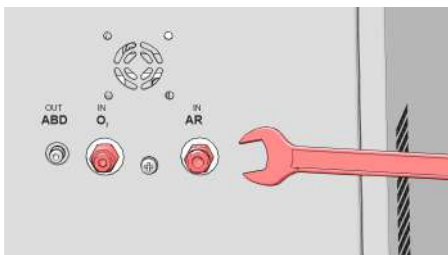


- ▶ Replace the sealing rings in the upper and lower part of the valve block.

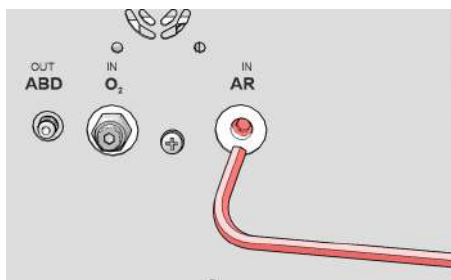
- ▶ Insert a new non-return valve.
- ▶ Assemble the valve block and screw on the top part.
- ▶ Connect hose 3 to the "main" connection of the valve block. Connect hose 4 to the "inlet" connection of the valve block.
- ▶ Connect the protective ground conductor to the side panel and close the side panel.
- ▶ Feed the water hose, absorber hose and transfer hose back out through the openings in the side wall and connect them to the respective modules and the fraction collector.
- ▶ Reconnect the ABD and install the ABD on the combustion tube.
- ▶ Open the gas supply at the shut-off valve.
- ▶ Connect the power cable on the basic module and switch on the module via the power switch.
- ▶ Perform a system leak test (→ "Checking the system for leaks" 71).
 - ✓ The digestion system is ready for operation again.

6.3.6 Replacing the particle filters in the gas inlets

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



- ▶ Switch off all devices in the system and disconnect the mains plugs.
- ▶ Switch off the gas supply at the shut-off valve.
- ▶ Disconnect the ABD from the base module, move it to the side, and disconnect the connections between the ABD and the base module (→ "Disconnecting the ABD from the combustion tube" 66).
- ▶ Pull the gas hose out of its connection on the rear of the basic module. To do so, press down the red ring and pull the gas hose out of the connection.
- ▶ Unscrew the gas connections with a 13 mm open-ended wrench.



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



- ▶ Insert and tighten the new particle filter.
- ▶ Screw in the gas connections and tighten them with an open-ended wrench. Connect the gas hoses.
- ▶ Reconnect the sample feeding module, if necessary.
- ▶ Switch on the gas supply.
- ▶ Connect the mains plug to the connection on the base module and switch the module on at the mains switch.
- ✓ The basic module is now ready for operation again.

6.3.7 Maintenance of the dosing syringes



CAUTION

Risk of crushing on the syringe pump

High forces are generated when the syringe drive is moved.

- Never reach into the slot of the dosing modules during operation.

Clean the syringes of the dosing modules if they are dirty. Replace the syringes if they are damaged or leaking.

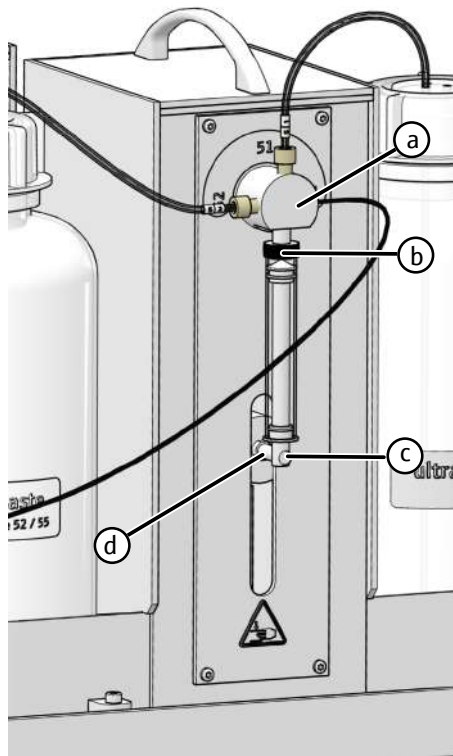
Empty syringe pumps and hoses

If the syringes are filled with solutions, you must first empty the syringes before removing them.

- ▶ Exit the multiWin software.
 - ✓ The syringes are emptied.
- ▶ Remove the hoses from the storage bottles for ultrapure water and absorber solution and wipe with a clean paper towel.
- ▶ De-energize the dosing modules. To do this, pull the power supply plugs out of the connections on the rear of the modules, wait a few seconds and reconnect the power supply units.
- ▶ Start the multiWin software.
 - ✓ Because the modules were de-energized, the syringe pumps are initialized. The residual liquid from the hoses for ultrapure water and absorber solution is drawn from the hoses into the syringe when the modules are initialized.
- ▶ Exit multiWin again.
- ▶ Repeat the process. De-energize the dosing modules and start multiWin.
- ▶ After initialization, remove the syringe from the pump module (see below).
- ▶ Exit multiWin.
- ▶ Pull the hoses out of the waste container and dispose of the waste solution.

- ✓ You can now remove the syringe pump. When decommissioning, you can now unscrew the hoses from the syringe pump valves. Wipe off residual liquids with a clean paper towel.

Syringe pump maintenance



- ▶ Switch on the digestion system and start multiWin.
 - ✓ When multiWin is started, the syringe drive moves to the position in which the syringe can be installed and removed.
- ▶ Unscrew the knurled screw (c) from the drive stem (d).
- ▶ Unscrew the dosing syringe (b) from the 3-way valve of the pump (a) and remove it.
- ▶ Clean the piston and syringe barrel.
- ▶ Screw the cleaned or a new dosing syringe (b) onto the valve (a).
- ▶ Carefully pull the piston downwards until the end of the piston is flush with the recess in the drive stem (d).
- ▶ Screw the piston finger-tight to the drive screw (d) using the knurled screw (c).
- ▶ Exit multiWin.
- ▶ Disconnect the electrical connections from the dosing modules, wait 5 seconds and reconnect the connections.
- ▶ Fill the bottles with ultrapure water and absorber solution and insert the hoses of the dosing modules into the corresponding bottles.
- ▶ Start multiWin and click the **[Initialize analyzer]** button.
 - ✓ The syringes are flushed. The dosing modules are ready for use.

6.3.8 Replacing tubes

The hoses are attached with FAST connectors or Fingertight connections. When changing the hoses, refer to the labels on the hoses, bottles and valve inlets of the dosing modules. This allows you to clearly assign the hoses. If labels are lost, replace them according to the illustrations.

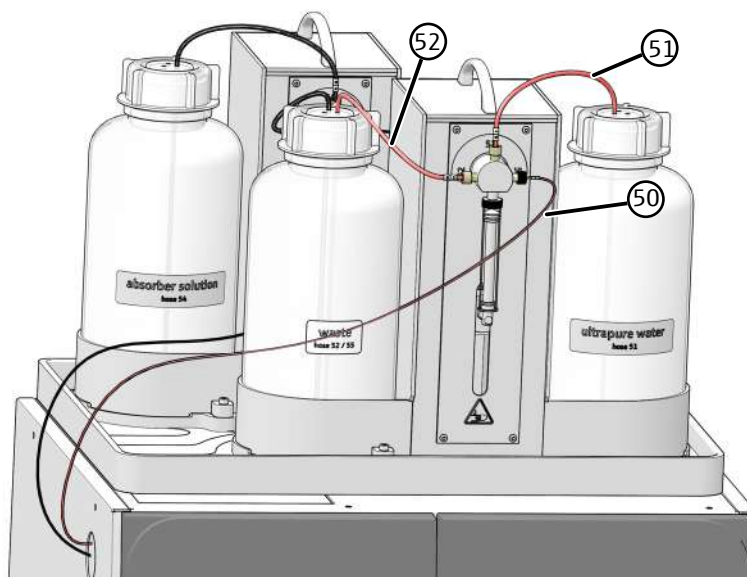


Fig. 30 Hoses on humidifier module

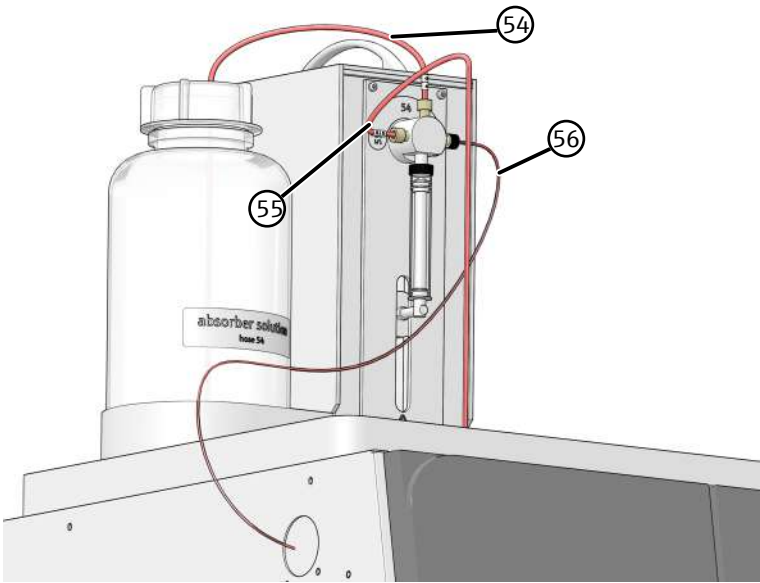


Fig. 31 Hoses on absorber module

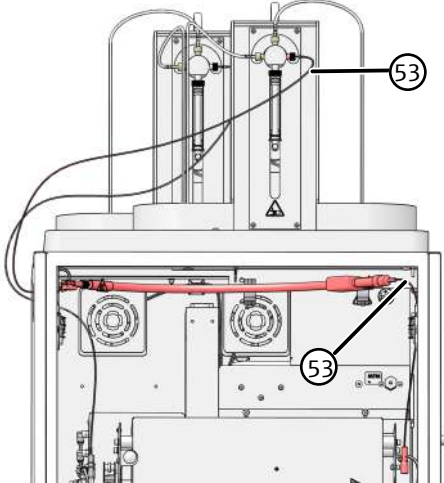


Fig. 32 Hose in the heated transfer line

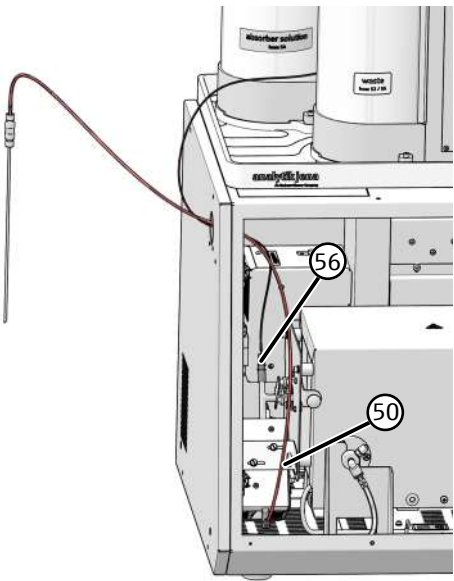


Fig. 33 Hoses on the T-connector

6.4 Maintenance of the ABD

The ABD must be disconnected from the digestion module for various maintenance work.

Particular care must be taken when attaching and removing the ABD to and from the combustion tube, as the combustion tube can easily break. Observe the following instructions precisely to minimize the risk of breakage.



Note:

Use the face spanner included in the scope of delivery to loosen and to firmly tighten the PEEK clamping rings at the sample port.

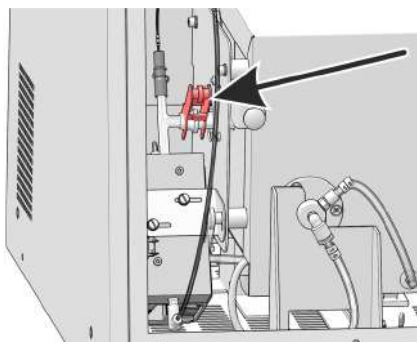
6.4.1 Disconnecting the ABD from the combustion tube



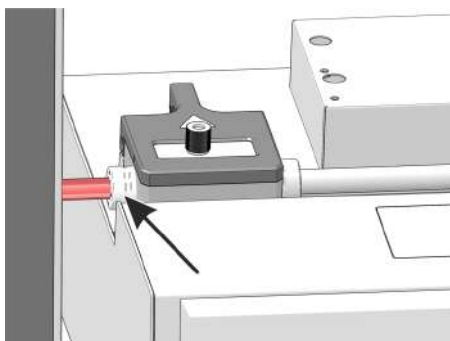
CAUTION

Risk of burns from hot combustion tube and screw connections

- Only carry out maintenance work on the ABD when it has cooled down.



- ▶ Switch off the base module and the ABD at the mains switch.
- ▶ Open the front doors of the base module. Remove the fork clamp between the T-connector and the combustion tube.

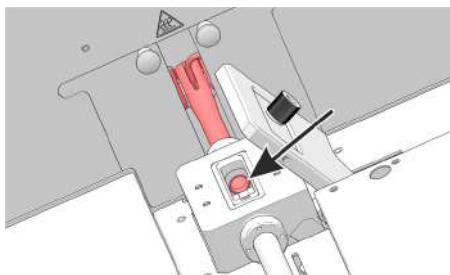


- ▶ Loosen the left clamping ring on the port using the face wrench provided.
- i** NOTICE! Risk of glass fracture! Proceed with caution. No pressure should be exerted on the combustion tube.

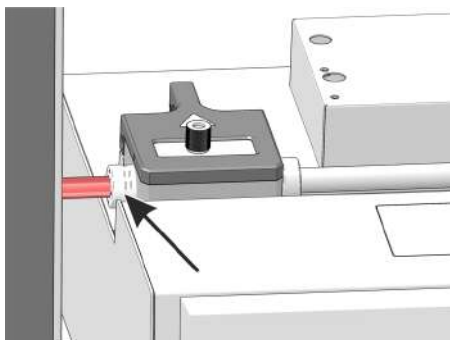
- ▶ Carefully pull the ABD to the right. The connecting piece of the combustion tube then slides out of the sample port.
- ▶ Pay attention to the length of the connections on the rear of the ABD (flame sensor; sampler cable, gas hose). These could easily tear off.
 - ✓ The ABD is separated from the combustion tube. You can lift the ABD off the guide.

6.4.2 Installing the ABD at the combustion tube

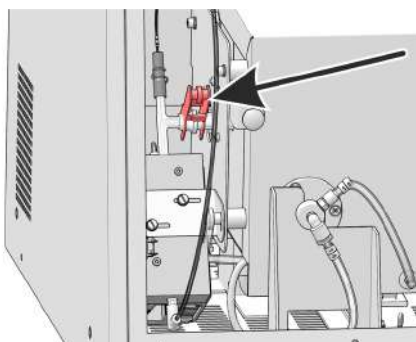
The combustion tube is already installed in the base module. The fork clamp is not attached to the spherical joint between the T-connector and the combustion tube.



- ▶ Place the ABD on the already mounted guide and carefully push it towards the base module until the combustion tube nozzle is resting against the stop in the sample port. The combustion tube can be pushed a few millimeters into the port by hand.
- ▶ Check the correct position of the combustion tube in the open port. The combustion tube must be flush with the port.



- ▶ Tighten the clamping ring on the sample port with the face wrench.



- ▶ Join the spherical joint of the T-connector and combustion tube and secure with the fork clamp.
 - ✓ The ABD is now installed on the combustion tube.

6.4.3 Replacing the septum and cleaning the sluice at the ABD



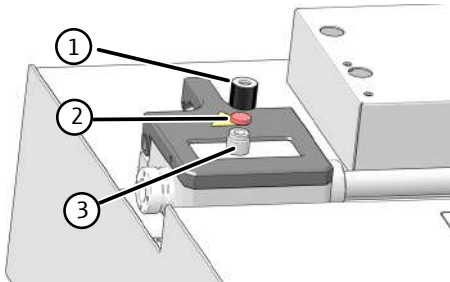
CAUTION

Crushing risk when closing the sample port!

High forces occur when closing the sample port of the ABD.

- Do not reach into the port area when closing.

Replace septum



- ▶ Unscrew the cap (1) from the injection port (3) and remove the used septum (2) from the cap.
- ▶ Insert a new septum into the cap.
- ▶ Place the cap on the injection port and tighten hand-tight.
 - ✓ The septum has been replaced.

Clean port

- ▶ Open the sample port using the "port" button on the ABD.
- ▶ Clean the inside of the port with ethanol-soaked cellulose. Allow the port to dry thoroughly afterwards.
- ▶ Close the port using the "port" button.
 - ✓ The port closes.

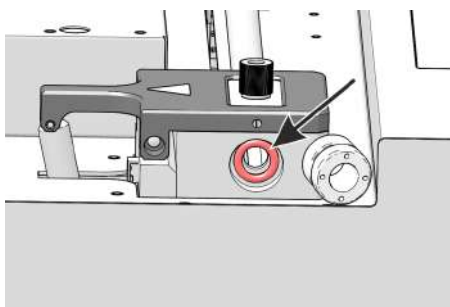
6.4.4 Replacing the seal on the sample port



CAUTION

Risk of burns from hot combustion tube and screw connections

- Only carry out maintenance work on the ABD when it has cooled down.



- ▶ Disconnect the ABD from the combustion tube (→ "Disconnecting the ABD from the combustion tube" 66).
- ▶ Push the ABD to the side.
- ▶ Unscrew the clamping ring completely from the sample port.
- ▶ Replace the sealing ring in the sample port.
- ▶ Loosely screw the clamping ring back into the sample port.
 - ✓ The sealing ring in the sample port has been replaced. You can now reinstall the ABD on the combustion tube (→ "Installing the ABD at the combustion tube" 67).

6.4.5 Cleaning and changing hooks



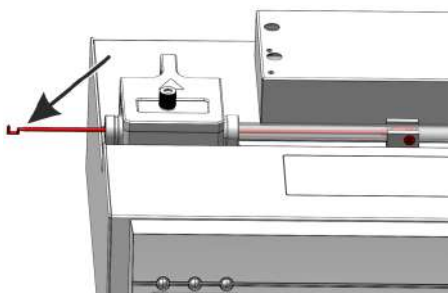
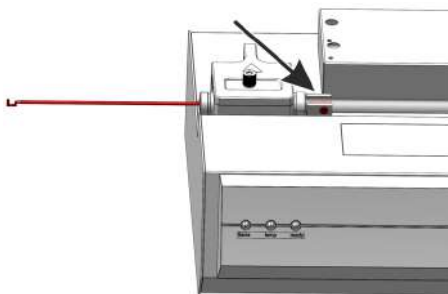
CAUTION

Risk of burns from hot combustion tube and screw connections

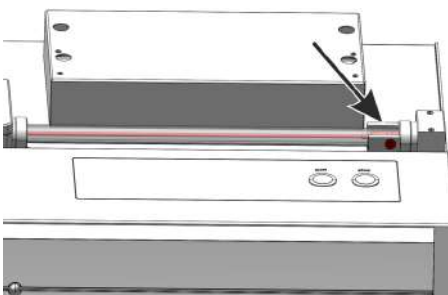
- Only carry out maintenance work on the ABD when it has cooled down.

The hook must be cleaned if it is dirty or replaced if it is damaged.

- ▶ Disconnect the ABD from the combustion tube.
- ▶ Switch off the ABD at the mains switch at the rear.
- ▶ Push the ABD to the side so that there is enough space to remove the hook. You can also lift the ABD from the guide.
- ▶ Pay attention to the length of the connections on the rear of the ABD (flame sensor; sampler cable, gas hose). These could easily tear off.
- ▶ Remove the protective cover from the guide tube.
- ▶ Push the magnetic coupling with hook carefully up to the stop towards the sample port (see arrow).
- ▶ Carefully pull the hook out of the magnetic coupling and then completely out of the sample port of the ABD.
 - ✓ You can now clean or replace the hook.



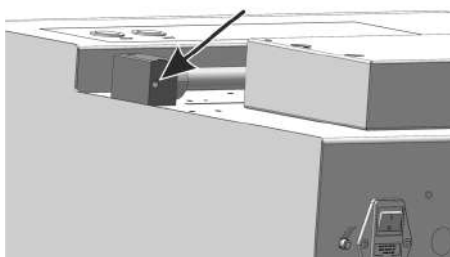
- ▶ Insert the cleaned hook or a new hook into the sample port.
 - i** NOTICE! Possible soiling. Wear gloves to avoid contaminating the hook.
- ▶ Carefully push the hook through the sample port until the coupling sleeve of the hook is pulled into the magnetic coupling. The hook must point upward when inserting.
- ▶ Slide the magnetic coupling to the right up to the stop at the block of the gas supply until the hook has been pulled in completely into the guide tube.
- ▶ Put the protective cover back onto the guide tube.
 - ✓ The hook is now installed. The ABD can now be re-installed on the combustion tube.



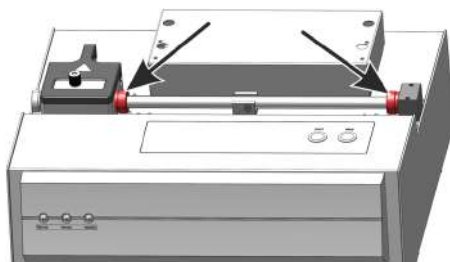
6.4.6 Cleaning or replacing the guide tube

If an autosampler has been fitted to the ABD, this must be removed prior to removing the guide tube (see user manual for MMS).

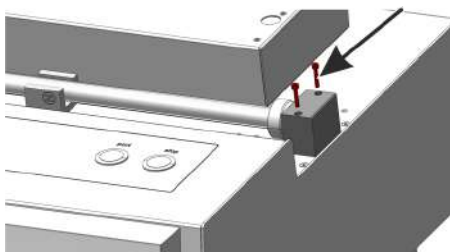
- ▶ Switch off the ABD at the mains switch.
- ▶ Disconnect the ABD from the combustion tube (→ "Disconnecting the ABD from the combustion tube" ☞ 66).
- ▶ Remove the hook from the guide tube (→ "Cleaning and changing hooks" ☞ 69).
- ▶ Unscrew the argon hose (arrow) from the gas supply block.



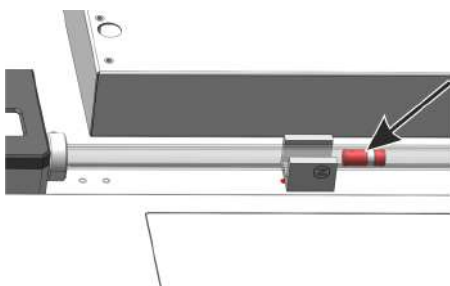
- ▶ Loosen the clamping rings on the right-hand side of the sample port and on the gas supply block using the face wrench.



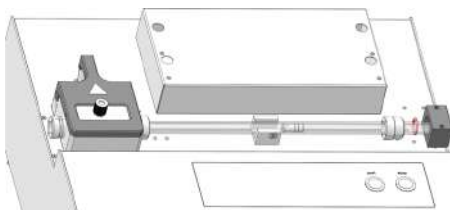
- ▶ Unscrew the screws on the gas supply block.
- ▶ Remove the guide tube with the block from the ABD.
- ▶ Remove the magnetic coupling from the guide tube.
- ✓ You can now clean or replace the guide tube.



- ▶ Push the magnetic coupling into the guide tube. The short piece of the magnetic coupling in front of the metal ring must face the block of the gas supply.



- ▶ Insert the guide tube into the block without tilting and screw on the block.
- ▶ Tighten the clamping rings again using the face wrench.
- ▶ Insert the cleaned hook or a new hook into the sample port of the ABD (→ "Cleaning and changing hooks" ☞ 69).
- ▶ Re-install the ABD on the combustion tube (→ "Installing the ABD at the combustion tube" ☞ 67).
- ✓ The system is now ready for operation again.



6.5 Checking the system for leaks



NOTICE

Destruction of the MFM by corrosive gases

Moisture can damage the internal MFM.

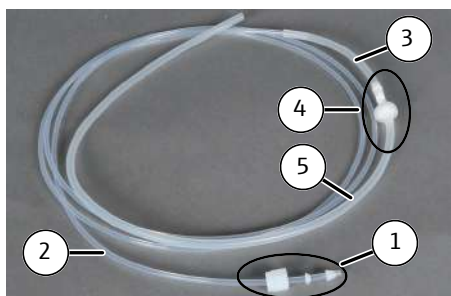
- Only use the flow test kit included in the scope of delivery to check the system for leaks.
- After sample digestion, wait at least 15 minutes before connecting to the internal MFM to ensure that there is no moisture in the sample path. Install the water trap and adapter in front of the flow meter.

The system tightness for the gas path can be checked from the inlet of the base module to the cannula. Use the flow check hose set included in the scope of delivery.

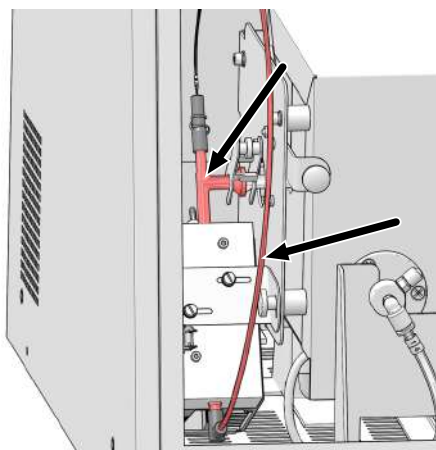
Requirements

- The base module is switched on.
- The carrier gas supply is open.
- The multiWin control and analysis software has been started.
- A method for the digestion system is activated and the furnace is heated to the set temperature of the method. The gas flow must be active during this time.

Procedure



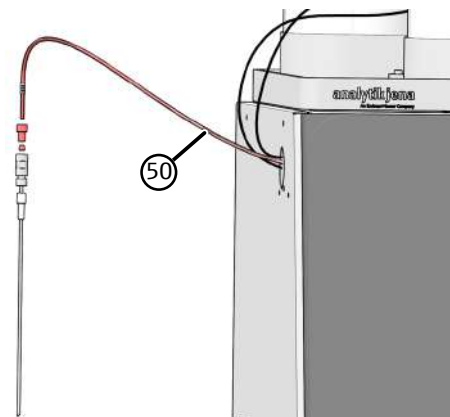
- ▶ Assemble the flow monitoring set in the following order:
 - Thread the screw cap, the seal and the sealing cone (1) onto the thin hose (2).
 - Connect the hose (2) with the other hose (3).
 - Attach the water trap and the adapter (4) to the hose (3).
 - Attach the hose (5) to the adapter.



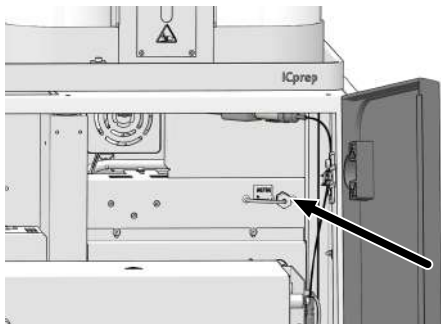
- ▶ Before connecting the hose set for the flow check, check the sample transfer hose from the T-connector to the cannula and the T-connector for aqueous residues.

Only carry out the flow check when the system is dry (residue-free).

i NOTICE! If drops are still visible in the transfer hose from the T-piece to the cannula or in the T-piece, plan for a longer waiting time with the active method and active gas flows.



- ▶ To check the system, loosen the Fingertight connection of the sample transfer hose from the coupling to the cannula.
- ▶ Attach the silicone hose (5) from the flow check set to the end of the transfer hose (50).



- ▶ Remove the "MFM in" screw connection on the cover of the control electronics in the basic module and connect the flow monitoring set (hose 2) (see arrow).
- ▶ Select the **System | Component test** menu item and in the **Component test | Flow** window, read off the current gas flow.
The target flow is the sum of the measured inlet gas flows (Main + Inlet), see "Display in the **Status analyzer** window" below.
- ▶ If the displayed flow deviates from the target flow by more than **5 %**, search for possible causes and rectify them. To do this, check the connections.
 - Hose connections (Fingertight connector and FAST connector)
 - Connection from the ABD to the combustion tube
 - Spherical joint between combustion tube and T-connector
 - Sealing rings and septum (→ "Maintenance of the ABD" 66)
- ▶ If this is not successful, contact AJ Service.
- ▶ After measuring the flow, remove the set and reconnect the hose (5) to the "MFM in" inlet.

Display in the window Status analyzer

The gas flows are set using the switches on the rear of the dosing modules or in the methods. The display in the **Status analyzer** window depends on the selected digestion method.

Switch position for single-phase digestion

Module	Switch	Position
humidifier module	Switch 1	1 – O ₂

Autosampler adjustment window Status analyzer

	Target	Description
MFC 1	200 or 300 mL/min	Main oxygen (hose 3) in the base module The value is set via switch 3 on absorber module .
MFC 2	50 ... 200 mL/min	Oxygen flow in the inlet (gas connection ABD) The value is set in the method on the Process tab (→ "Create methods" 43).
MFC 3	-	Is in idle state

Switch position for two-phase digestion

Module	Switch	Position
humidifier module	Switch 1	0 – Ar+O ₂

Autosampler adjustment window Status analyzer

	Target	Description
MFC 1	200 or 300 mL/min	Main oxygen (hose 3) in the base module The value is set via switch 3 on absorber module .
MFC 2	-	Is in idle state
MFC 3	50 ... 200 mL/ min	Argon flow in the inlet (gas connection ABD) The value is set in the method on the Process tab (→ "Create methods" ⓘ 43)

See also

📘 Maintenance of the ABD [▶ 66]

6.6 Recommissioning after emergency shutdown or shutdown due to gas pressure fault



CAUTION

Risk of chemical burns

In the event of an emergency shutdown, the hoses and the combustion tube may be contaminated with acidic solutions, e.g. hydrofluoric acid.

- Check the hoses before putting the digestion system back into operation.
- Wear appropriate protective clothing.

Recommissioning after emergency shutdown

- ▶ Check whether there is moisture in the hoses or in the combustion tube. These could be acidic samples.
- ▶ If necessary, carefully disconnect the sample transfer line from the outlet of the cooling block and collect any escaping liquid.
- ▶ Remove the fork clamp and disconnect the T-connector from the combustion tube.
- ▶ Remove the combustion tube (→ "Removing and cleaning the combustion tube" ⓘ 56).
- ▶ Thoroughly rinse the combustion tube with ultrapure water and then dry, e.g. in a drying cabinet.
- ▶ Reinstall the combustion tube (→ "Installing the combustion tube" ⓘ 57).
- ▶ Clean or replace the hoses.
 - ✓ The digestion system is now ready for operation again.



Recommissioning after gas pressure fault

CAUTION

Risk of injury from hot, corrosive vapors

Hot and corrosive vapors can escape quickly when the sample port on the ABD is opened and cause burns and chemical burns.

- Never open the sample port on the ABD in the event of a gas pressure error.
 - Always search for possible system closures starting with the sample cannula.
 - Allow the system to cool down sufficiently.
-

If there is excess pressure in the system, the gas flow is interrupted and an error message appears (206 Gas pressure fault). Locate the closure in the system starting at the cannula.

- ▶ Unscrew the cannula from the hose.
- ▶ Reinitialize the system and check whether the gas pressures are now correct.
- ▶ If this is not successful, first remove the sample transfer hose from the T-piece and then the T-piece itself and check the gas pressure. Then also check the hoses to the combustion tube.
- ▶ Clean the hoses, T-piece, cannula and combustion tube and reassemble the system.

7 Troubleshooting

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

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



CAUTION

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

7.1 Remediating software message faults

Communication problems between the hardware and the software can often be remedied by a basic initialization of the measuring system (→ "Initializing the basic module and the system components" 78).



NOTICE

Communication fault due to wrong USB cable

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

Error code	Error message/cause	Remedy
1	No response from firmware!	
	Basic module not switched on	Switch on the basic module
	Basic module disconnected from PC	Check the connection between the basic module and the PC
	Incorrect port selected	Check which port the device is plugged into on the PC Select another interface (Configuration Interface menu item) Initialize
2	Serial port not available!	
3	Serial port not reachable!	
	Communication problems	Disconnect the USB connection between the basic module and the PC and reconnect after approx. 10 s Initialize
7	Operating system errors: Unauthorized access	
	Undefined multiWin end	Exit the software and switch off the device Detach the USB cable and re-connect after approx. 10 s Restart the operating system (PC)

Error code	Error message/cause	Remedy
		Switching the device on Restart the software
12	Signal echo received, check port selection	
	Incorrect port selected	Check port selection
14	Data transfer interrupted	
	No data transfer for 10 s	Check port selection
17	Incorrect interface protocol ID	
	Error after update (the program versions of the firmware and multiWin do not match)	Update required
20	Timeout: InitEnd	
	Timeout during initialization	Initialize
21	Timeout: StatusBusy	
	Timeout during operation (device not ready to measure)	Acknowledge message Initialize
22	Timeout: End	
	Timeout when exiting multiWin	Acknowledge message Initialize
23	Timeout: StopEnd	
	Timeout during measurement cancellation	Acknowledge message Initialize
50	Firmware reset	
	Internal computer (firmware) restarted	Acknowledge message Initialize
61	Command from PC incomplete	
62	Command from PC without STX	
64	Command from PC CRC error	
65	Command from PC invalid	
66	Command from PC invalid MEAS command	
	Communication error	Acknowledge message Initialize
200	Gas box: No connection	
	Communication error	Acknowledge message Initialize
201	Gas box: Error when setting the target flow	
	Communication to gas box interrupted	Acknowledge message Initialize
202	Gas box: Conversion error 1	
203	Gas box: Conversion error 2	
204	Gas box: Conversion error 3	
205	Gas box: Conversion error 4	
	Communication faulty (readout of flows from gas box faulty)	Acknowledge message Initialize
206	Gas pressure error	

Error code	Error message/cause	Remedy
	Overpressure in the analysis system due to blocked gas paths	 WARNING! Extreme caution is required during system overpressure! Never switch off a device subject to overpressure! Otherwise there is a risk of injury for the operating personnel and the device system will be damaged. For recommissioning, observe the instructions in the relevant section (→ "Recommissioning after emergency shutdown or shutdown due to gas pressure fault"  73)
220	Sampler: no connection	
	Communication interrupted after autosampler detection during initialization	Acknowledge message Initialize
222	Boat: Broken	
	Boat defect during removal from the combustion tube (only if boat sensor is used)	Remove the broken boat from the system Initialize
223	Sampler: Incorrect peak value	
	No syringe inserted	Insert syringe in autosampler Initialize
	Dosed volume in the method greater than inserted syringe volume	Adjust dosed volume or insert suitable syringe Initialize
	A method for dosing liquids is to be activated and the gripper and solids track are still inserted	Insert sample rack for liquid samples Insert syringe Initialize
224	Sampler: Incorrect gripper	
	No gripper inserted	Insert gripper in autosampler Initialize
	The method for solids is to be enabled and the syringe and sample rack for liquid samples are still inserted	Insert solids rack Insert gripper Initialize
226	Sampler: Runtime exceeded	
	Completion message for the autosampler movement takes too long (autosampler faulty)	Record log files Inform customer service department
230	ABD: No connection	
	Communication disrupted after ABD was detected during initialization	Acknowledge message Initialize
231	ABD: Runtime exceeded	
	Completion message for the ABD movement takes too long	Check that the flame sensor is properly applied and connected Record log files Inform customer service department
232	Flame sensor error	
	Flame sensor calibration failed	Record log files Inform customer service department
260	Sample handling missing	
	No sample feeding module detected	Connect at least one sample feeding module Initialize

Error code	Error message/cause	Remedy
300	Temperature controller: No connection	
	Communication error	Acknowledge message Initialize
304	Temperature controller: Communication error	
	Temperature cannot be set	Acknowledge message Initialize

7.2 Initializing the basic module and the system components

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

During standard initialization, only the system components that were active before multiWin was last shut down and the switch position of the dosing modules are queried. The last active method is then loaded.

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

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

Performing the basic initialization

The basic initialization always takes place when the **Device - edit** is opened and closed with **[OK]**:

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

Standard initialization

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

7.3 Device faults on the basic module

Error	Possible cause	Remedy
Furnace does not heat	Thermocouple connector not connected	Connect the connector (→ "Removing and installing the combustion furnace"  27)
	Temperature set incorrectly in the software	Check temperature configuration in the method
	No method loaded	Loading a method
	Malfunction in power supply	Switching the device on Check the internal fuse

Error	Possible cause	Remedy
Furnace temperature is outside tolerance limits or target temperature is not reached		Check the connection between the basic module and the PC
	Malfunction in the internal electronics	Inform customer service department
	Temperature control faulty Electronics error	Inform customer service department
Process gases (inlet flow) not supplied	Gas supply not connected	Connect the gas supply
	Primary gas pressure too low	Set the primary gas pressure at the delivery point to 600 kPa (6 bar)
	Gas supply leaking	Check gas supply
	No method loaded	Loading a method
	Gas box faulty	Inform customer service department
Target flow at the outlet to the fraction collector too low	Connection between hose, angled adapter and combustion tube not correct	Check connection and ensure correct fit at the connection points
	Septum in the injection port of the ABD is positioned incorrectly or is leaking	Check position of the septum, insert new septum if necessary
	Connection between combustion tube and ABD leaking	Check the seal of the ABD Check alignment of combustion tube and ABD
		Tighten screw finger-tight

7.4 Analytical problems in the basic module

Error	Possible cause	Remedy
Low results independent of detection	Dosing error	Check dosing by checking the autosampler
	System leaking	Check system tightness
	Temperature set too low	Check temperature configuration in the method
	Sample loss due to vaporization or spillage	Keep liquid samples closed. If possible, used a cooled autosampler. Check sampler function for solids
	Post-combustion period insufficient	Particularly for solids, a post-combustion period of at least 120 s must be set
	Sooting in the system	Clean or replace sooty components
Carryover	Inadequate sampler component rinsing	Rinse dosing syringes adequately prior to sampling
	Combustion tube not rinsed adequately	Rinse combustion tube adequately with clean solvent, i.e. blank measurements until values are constant
	Contamination of sample port of the ABD	Replace septum Clean the port
	Insufficient cooling of the sample port of the ABD	Check cooling, if necessary inform Service
Scattering measurements	Dosing faulty	Check dosing process
	Combustion tube contaminated or severely crystallized	Clean or replace combustion tube

Error	Possible cause	Remedy
Blank values too high	Quartz boat not tempered with carrier material	Temper fresh carrier material with quartz boat (blank measurement)
	Contamination of the port on the ABD	Replace septum
		Clean the port

8 Transport and storage

8.1 Transport

When transporting the device, observe the safety instructions in the "Safety instructions" section.

Avoid the following during transport:

- Impact and vibration
Risk of damage due to shock, impact or vibration!
- Large temperature fluctuations
Risk of condensation!

8.1.1 Moving the device in the laboratory



CAUTION

Risk of injury during transport

Dropping the device poses a risk of injury and damage to the device.

- Proceed carefully when moving and transporting the device. Two persons are required to lift and carry the device.
- Grip the device firmly at the bottom with both hands and lift it simultaneously.

Observe the following when moving the device within the laboratory:

- Insufficiently secured components pose a risk of injury!
Before moving the device, remove all loose parts and disconnect all connections from the device.
- For safety reasons, two persons are required to transport the device, one person on each side of the device.
- As the device does not have carrying handles, grip the device firmly with both hands at the lower end. Lift the device simultaneously.
- Observe the guide values and adhere to the legally mandated limits for lifting and carrying loads without auxiliary means.
- Observe the installation conditions at the new location.

8.1.2 Preparing the device for transportation and storage



WARNING

Risk of damage to health due to improper decontamination

- Decontaminate the device professionally and document the cleaning measures before returning the device to Analytik Jena.
 - The customer service department will send you the decontamination declaration when you register the return.
-



CAUTION

Risk of burns from the hot furnace

- Only remove the combustion tube and the combustion furnace when they are cold. Allow the device to cool sufficiently!



CAUTION

Risk of injury

Glass and ceramic parts can break easily. Therefore there is a risk of injury when handling them.

- Handle glass and ceramic parts particularly carefully.



NOTICE

Risk of device damage due to unsuitable packaging material

- Only transport the device and its components in the original packaging.
- Empty the device completely and attach all transport locks before transporting the device.
- Add a suitable desiccant to the packaging to prevent damage from moisture.

Packing the base module and dosing modules

- ▶ Empty the syringe pumps and hoses of the dosing modules (→ "Maintenance of the dosing syringes" 63).
- ▶ Switch off the base module and system components and allow to cool down.
- ▶ Switch off the gas supply and disconnect the mains plug from the mains socket.
- ▶ Remove the bottles from the dosing modules, empty and dry them.
- ▶ Disconnect the ABD from the combustion tube (→ "Disconnecting the ABD from the combustion tube" 66).
- ▶ Remove all connections on the rear of the base module. Pull the dongle out of the connection.
- ▶ Remove the combustion tube (→ "Removing and cleaning the combustion tube" 56).
- ▶ Remove the T-connector from the Peltier cooling block (→ "Remove and clean the T-connector" 60) and remove the cooling block (→ "Removing and installing the Peltier cooling block" 31).
- ▶ Remove the dosing modules and the heated transfer line for the ultrapure water supply. Removal is carried out in the reverse order of installation (→ "Installing dosing modules and transfer line" 28).
- ▶ Remove the combustion furnace (→ "Removing and installing the combustion furnace" 27).
- ▶ Unscrew the guide plate of the ABD from the base of the base module.
- ▶ Pack open hose ends in protective bags and secure them with adhesive tape.
- ▶ Position the top cover and secure it with adhesive tape.
- ▶ Fix the doors on the right-hand side of the device with adhesive tape.

- ▶ Pack components in original packaging. The glass components in particular must be packed safely against breakage.

Packing the fraction collector

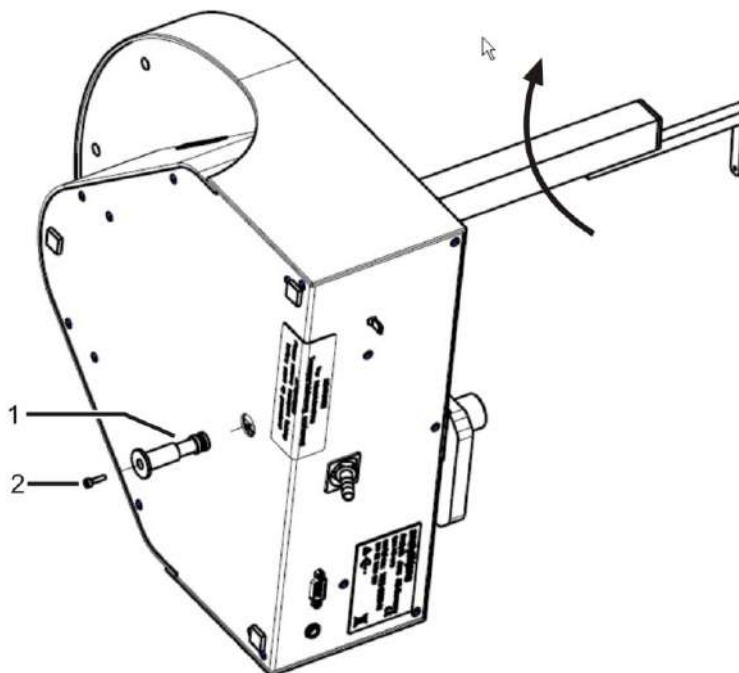


Fig. 34 Secure the fraction collector for transportation

1 Transport lock

2 M3x12 screw

- ▶ Loosen the fastening screw on the cannula guide on the autosampler arm and pull out the cannula with tube.
- ▶ Remove the sample tray.
- ▶ For the fraction collector ER: Remove and dry the cannula rinse.
- ▶ Turn the device on its side and set it down safely.
- ▶ Turn the autosampler arm clockwise as far as it will go.
 - ✓ The drives are in the correct position.
- ▶ Slide the transport lock into the opening of the bottom plate up to the stop.
- ▶ Fasten the transport lock with the screw and the hexagon socket key supplied.
- ▶ Pack the device in its original packaging.

Packing the ABD and Multi Matrix Sampler

Information on packing the devices can be found in the corresponding operating manuals.

8.2 Storage



NOTICE

Risk of device damage due to environmental conditions

Environmental influences and condensation can destroy individual components of the device.

- Only store the device in air-conditioned rooms.
- Ensure that the atmosphere is free of dust and corrosive vapors.

If the device is not installed immediately after delivery or not required for longer periods, it should be stored in its original packaging. A suitable desiccant should be added to the equipment to prevent damage from moisture.

The requirements for the climatic conditions of the storage location can be found in the specifications.

9 Specifications

9.1 Technical data for the ICprep

General characteristics	Name	ICprep	
	Digestion principle	Pyrohydrolysis with subsequent thermal oxidation	
	Digestion temperature	700 to 1100 °C	
	Recommended digestion temperature	1050 °C	
	Process control	multiWin control and analysis program	
Sample feed with ABD	Liquids	Injection of liquids via injection port with septum in quartz boats	
	Solids	Solid samples in quartz boats	
Sample volume	Liquids	10 to 100 µl	
	Solids	10 to 100 mg	
Gas supply	Process gases	Inlet pressure	Consumption
	Argon ≥ 4.6 (halogen- and hydrocarbon-free)	600 kPa (6 bar)	100 – 200 mL/min
	Oxygen ≥ 4.5	600 kPa (6 bar)	Pyrolysis: 300 ml/min Post-combustion: 400 ml/min
Electrical variables	Power supply	110 to 240 V AC	
	Overvoltage category	II	
	Frequency	50/60 Hz	
	Fuses	T 10 A H (2x)	
	Typical average power consumption	1000 VA	
	PC interface	USB	
Ambient conditions	Temperature during operation	+20 to +35 °C	
	Humidity during operation	Max. 90 % at 30 °C	
	Air pressure	0.7 to 1.06 bar	
	Max. insert height	2000 m	
	Temperature during storage	+15 to +55 °C	
	Humidity during storage	10 to 30 % (use desiccant)	
	Protection class	I	

	Protection class of the housing	IP 20
Mass and dimension of base module	Mass	25 kg
	Dimensions (W x H x D)	530 x 470 x 560 mm
Dosing modules	Dosing modules	humidifier module - dosing of ultrapure water absorber module - dosing of absorber solution
	Mass (total)	9.7 kg
	Dimensions (W x H x D)	490 x 370 x 320 mm
	Storage bottles	Bottles for ultrapure water, absorber solution, waste 2 L each
	Operating voltage	24 V DC, via external power supplies
	Voltage, frequency external power supply units (a separate power supply unit for each module)	100 to 240 V, 50/60 Hz (autosensing)
	Power consumption per module	36 VA
Fraction collector AS vario/AS vario ER	Dimensions (W x D x H)	350 x 400 x 470 mm
	Mass	15 kg
	Sample tray	100 sample tubes (15 mL, height 119 mm, Ø 16 mm)
	Operating voltage	24 V DC via external power supply
	Power supply, external power supply unit	100 to 240 V, 50/60 Hz (autosensing)
	Power consumption	50 VA
Minimum requirements for the computer	Graphics resolution	1280 x 1024
	CD/DVD drive	To install the software
	Interface	1 x USB 2.0
	Operating system	Windows 8.1, Windows 10 or Windows 11

9.2 Guidelines and standards for ICprep dosing modules

Directives/regulations	The humidifier module and absorber module products comply with the following European directives: 2006/42/EC – Machinery Directive 2014/30/EU – EMC Directive 2011/65/EU – RoHS Directive
Standards	The following harmonized standards or normative documents have been applied to the humidifier module and absorber module products: EN 61010-1

EN 61326-1

EN IEC 63000

Directives for the People's Republic of China

The device contains regulated substances (according to directive GB/T 26572-2011). Analytik Jena warrants that these substances will not leak when used as intended for the next 25 years and therefore do not pose a risk to the environment or health during this period.

List of figures

Fig. 1	Model ICprep automatic	12
Fig. 2	Base module (without front doors)	13
Fig. 3	LED display for operational readiness	13
Fig. 4	Connections on the rear of the device	14
Fig. 5	Electrical connections in the interior of the base module	14
Fig. 6	Electrical connection for the heated transfer line	15
Fig. 7	Electrical connection for Peltier cooling block.....	15
Fig. 8	Quartz combustion tube for standard applications.....	16
Fig. 9	Ceramic combustion tube for samples with salt loads.....	16
Fig. 10	FAST connector	17
Fig. 11	Fingertight screw connection	17
Fig. 12	Peltier cooling block with T-connector	18
Fig. 13	Dosing modules with storage bottles	19
Fig. 14	Connections and switches on the rear of the dosing modules.....	20
Fig. 15	Automatic Boat Drive (ABD)	21
Fig. 16	MMS 5100	21
Fig. 17	Structure of the fraction collector	22
Fig. 18	Functional diagram of ICprep	23
Fig. 19	Space requirements for ICprep automatic digestion system	25
Fig. 20	Connections on the rear of the ABD	33
Fig. 21	Transport lock on the fraction collector.....	34
Fig. 22	Cannula rinsing on the fraction collector.....	36
Fig. 23	Install the ceramic combustion tube	37
Fig. 24	Quartz boat with correctly inserted quartz fleece	51
Fig. 25	Boat with quartz container	51
Fig. 26	Boat with retainer for filters.....	51
Fig. 27	Attach the cannula to the stand.....	53
Fig. 28	FAST connector, angled	59
Fig. 29	Replacing the Fingertight connection.....	60
Fig. 30	Hoses on humidifier module	64
Fig. 31	Hoses on absorber module	65
Fig. 32	Hose in the heated transfer line.....	65
Fig. 33	Hoses on the T-connector	65
Fig. 34	Secure the fraction collector for transportation	83