

Operating Manual

PlasmaQuant 9100 (Elite) High-Resolution Array ICP-OES



Manufacturer Analytik Jena GmbH
Konrad-Zuse-Straße 1
07745 Jena / Germany
Telephone: +49 3641 77 70
Fax: +49 3641 77 9279
E-mail: info@analytik-jena.com

Technical Service Analytik Jena GmbH
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>

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1 Basic information

1.1 About this user manual

Contents

This user manual describes the following device models:

- PlasmaQuant 9100
- PlasmaQuant 9100 Elite

In this following, these models are collectively referred to as PlasmaQuant 9100. Any differences between the models are explained in the corresponding section.

This device is intended for operation by qualified specialist personnel observing this user manual.

This user manual provides information about the design and operation of the device and provides operating personnel with the necessary know-how for the safe handling of the device and its components. Furthermore, this user manual includes information on the maintenance and care of the device as well as notes on potential causes for malfunctions and their repair.

Conventions

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

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).
- Buttons are indicated by square brackets (e.g., [OK]).
- 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. The warnings are always placed before an action.



WARNING

Indicates a potentially hazardous situation which can cause death or very serious (possibly permanent) injury.



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 ICP Emission Spectrometer (ICP-OES) is used in a chemical analysis laboratory for the analysis of liquid - mainly aqueous - samples to determine the concentrations of up to 75 elements up to the trace range.

The device and its components may only be used for the analyses listed in the user manual. Only this specified use is regarded as the intended use, ensuring the safety of the user and the device.

This device is not suitable for solutions containing hydrofluoric acid if the nebulizer or spray chamber is made of glass or quartz. Use hydrofluoric acid-resistant components for this purpose. Take special precautions when working with organic solvents. In addition to apparatus-related and methodical aspects, fire and health protection regulations for any organic solvents must be observed.

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 of a hazardous location	<ul style="list-style-type: none"> At the exhaust vent: Warning against optical radiation. If no exhaust air hose is connected, looking indirectly (by mirror) into the radiation of the plasma is possible. In the sample chamber: Warning of moving parts; warning of hot torch In the plasma compartment: Warning against hot surfaces At the power inlet: Caution when connecting the media (gases, cooling water and power)
	Warning against hot surfaces	<ul style="list-style-type: none"> In the plasma compartment: Warning against hot surfaces. Immediately after the plasma is extinguished, the surface of the plasma compartment, and in particular the torch and plasma chamber window components, and the induction coil, are hot. Risk of burns!
Notice symbol	Meaning	Comment
	Observe the user manual.	<ul style="list-style-type: none"> At the power switch: Before starting work, read the user manual.
	Always disconnect the power before opening the device	<ul style="list-style-type: none"> On the side panel at the power inlet: Before opening the device cover, switch off the device and unplug the power plug.
	People's Republic of China only:	This device contains controlled substances. Analytik Jena guarantees that these substances will not escape the device in the next 25 years if operated in accordance with its intended purpose.

The following notice sign is attached to the rear of the device:

Caution! Power is still connected even when the device is switched off!

Unplug the power cable before opening!

Only connect or disconnect accessories when the device is switched off!

2.2 Requirements for the operating personnel

The device must only be operated by qualified specialist personnel instructed in the use of the device. This instruction also include teaching the contents of this user manual and of the user manuals of the connected system components. We recommend training by qualified employees of Analytik Jena or its representatives.

In addition to the safety instructions in this user manual, the general applicable safety and accident prevention regulations of the respective country the device is operated in must be observed and adhered to. The operator must ensure the latest version of these regulations.

The user manual must be accessible to the operating and service personnel.

2.3 Safety instructions, transport and commissioning

Incorrect installation can create serious hazards. This may result in electric shock and explosion if the gases are not connected correctly.

- Only the Analytik Jena GmbH customer service or specialist personnel trained and authorized by them is allowed to install and commission the device and its system components.
- Unauthorized assembly and installation is not permitted.

Insufficiently secured components pose a risk of injury.

- During transport, secure the device components as specified in these operating instructions.
- Loose parts must be removed from the system components and packed separately.

To prevent personal injury, the following must be observed when moving the device in the laboratory (lifting and carrying):

- Use a lift truck to transport the device.
- Four people positioned on the sides of the device are required to carry the device by four firmly mounted carrying handles.
- Risk of damage to health due to improper decontamination! Perform a professional and documented decontamination of the device before returning it to Analytik Jena. The decontamination report is available from Service when registering the return. Service must refuse acceptance of contaminated devices. The sender may be liable for damage caused by inadequate decontamination of the device.

2.4 Safety instructions: during operation

2.4.1 Basic safety information for operation

The operator must make sure that the device and its safety equipment is in sound condition each time before starting up the device. This applies in particular after each modification or extension of the device or its repair.

Observe the following:

- The device may only be operated if all items of protective equipment (e.g. covers in front of electronic components) are in place, properly installed and fully operational.
- The sound condition of the protection and safety equipment must be checked regularly. Any defects must be corrected as soon as they occur.
- Protective and safety equipment must never be removed, modified or switched off during operation.
- Modifications, conversions and extensions to the device are only permitted after consultation with Analytik Jena. Unauthorized modifications can jeopardize the device's operational safety and may lead to limitations regarding the warranty and access to customer service.
- During operation, free access to the connections, the power switch and the manual plasma deactivation switch on the left housing panel must be ensured.
- The ventilation equipment on the device must be in good working condition. Covered ventilation grilles or slots etc. may cause the device to break down or may cause damage to it.
- Caution when handling glass components. Risk of broken glass and therefore risk of injury!
- Ensure that no liquid enters the interior of the device, for example at cable connections. There is a danger of electric shock.
- During operation, there is a risk of crushing at the hose pump. Long hair and baggy clothing can become caught in the pump and drawn in. Wear suitable hair protections and tight-fitting clothing.

2.4.2 Safety instructions – protection against explosion and fire

The device may not be operated in an explosive environment.

Smoking or handling open flames are prohibited in the room in which the device is operated!

2.4.3 Safety instructions – electrical equipment

Lethal voltages may occur in the device! Contact with live components may cause death, serious injury or painful electrical shock.

- Work on the electronics may only be carried out by the customer service of Analytik Jena and specially authorized technicians.
- The power plug must be connected to a proper power outlet to ensure that the device meets protection class I (ground connector). The device may only be connected to power sources whose nominal voltage is the same as that on the rating plate. The protective effect must not be invalidated by the use of an extension line without a protective conductor.
- The basic module and the system components may only be connected to the mains when they are switched off.

- Electrical connection cables between the basic module and the system components may only be connected or disconnected when the device is switched off.
- Before opening the device, the device must be switched off via the main switch and the power plug must be disconnected from the power outlet!


2.4.4 Hazards caused by plasma operation

Plasma is extremely hot (up to 10000 K), and it emits electromagnetic as well as UV radiation. The induction coil operates at 1500 V RMS and 40.68 MHz. High-frequency radiation and UV radiation can cause serious injuries to skin and eyes. Contact with the torch (plasma torch) shortly after operation will cause skin burns. An electrical discharge may occur even across larger distances, causing fatal injuries, electrical shock and skin injuries.

Observe the following:

- To ensure safe plasma torch (torch) operation, the plasma must not be ignited unless the following conditions are met:
 - The plasma compartment door is closed.
 - The torch is in working position.
 - Sufficient cooling is supplied.
 - The extractor device is connected and switched on.
 - Argon supply is ensured.

Note: The aforementioned components are backed up by hardware safety circuits. Unless the reliable functioning of these components is ensured, the plasma will not be ignited, or it will be extinguished automatically if a component reports a malfunction.

- The safety circuits must not be bridged.
- Before opening the plasma compartment door, extinguish the plasma in the ASPECT PQ software. To do this, click on the  button in the program toolbar.
- Wait for the compartment to cool down for at least 5 min and avoid touching any hot parts of the torch or its surroundings immediately after extinguishing the plasma.

2.4.5 Behavior in case of ring plasmas

The manual plasma deactivation switch is located on the left-hand side of the device (red).

In the following situations, press the deactivation switch immediately to prevent the torch from melting:

- The plasma produces loud noises (rattles).
- The shape of the plasma changes considerably and a shining ring is visible on the inside of the coil.
- Parts of the torch begin to glow red-hot.

2.4.6 Safety instructions on the formation of ozone and toxic vapors

The interaction between the UV radiation from the torch and the surrounding air results in the formation of a high concentration of toxic gases such as ozone and nitrogen oxides. Additionally, toxic byproducts may escape from the samples and during sample processing.

Observe the following:

- The device may only be operated when the exhaust unit is activated.
- The exhaust unit must be switched on before igniting the plasma.

2.4.7 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 operating gases must have the required purity.
- Work on compressed gas containers and systems may only be carried out by individuals with specialist knowledge and experience in compressed gas systems.
- Compressed air hoses and pressure reducers may only be used for the assigned gases.
- Pipes, hoses, screw connections and pressure reducers for oxygen must be kept free from grease.
- Check all pipes, hoses and screw connections regularly for leaks and externally visible damage. Repair leaks and damage without delay.
- Shut off the gas supply to the device prior to any maintenance and repair work on the compressed gas containers.
- After successful repair and maintenance of the components of the compressed gas containers or system, the device must be checked for proper operation prior to recommissioning.
- Unauthorized assembly and installation are not permitted!

2.4.8 Handling of samples, auxiliary and operating materials

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

When handling hazardous substances, the locally applicable safety instructions and instructions in the safety data sheets from the manufacturers of the auxiliary and operating materials must be complied with.

- Cleaning with hydrofluoric acid must be carried out in an exhaust chamber. The operator must wear a rubber apron, gloves and a face mask when handling hydrofluoric acid.
- When measuring material containing cyanide, ensure that no hydrogen cyanide can be generated the waste bottle, i.e. the waste solution must not be acidic.
- Ensure that all residual liquid from the nebulizer and the sampler is emptied into the waste bottle supplied.
- The operator is responsible for ensuring that waste materials such as drained coolant and residual liquid from the waste bottle are disposed off in an environmentally responsible manner and according to local regulations.
- Flammable and health-hazardous organic solvents such as toluene, ethanol or methanol can be used in the operation of the device. In there is uncertainty about a solvent, it may only be used when the manufacturer has confirmed that there is no danger to safety.

2.4.9 Safety instructions on cleaning and decontamination measures

Observe the following:

- The operator is responsible for carrying out suitable decontamination should the device become contaminated externally or internally with dangerous substances.
- Splashes, drops or larger liquid spillages should be removed using an absorbent material such as cotton wool, laboratory wipes or cellulose.
- For biological contamination, wipe the affected area with a suitable disinfectant, such as an Incidin Plus solution. Then wipe the cleaned areas so that they are dry.
- The only suitable cleaning method for the housing is wipe disinfection. If the disinfectant has a spray nozzle, apply disinfectant to a suitable cloth before using it on the device.
Particular care must be taken if infectious materials are analyzed with the device, as the device cannot be decontaminated as a single unit.
- Before using a cleaning or decontamination procedure other than that prescribed by the manufacturer, the user is required to check with the manufacturer that the intended procedure will not damage the device. Safety labels attached to the device must not have methanol applied.

2.5 Safety instructions for maintenance and repair

The device is generally maintained by the customer service department of Analytik Jena or specialist personnel trained and authorized by them.

Unauthorized maintenance can damage the device. For this reason, only the activities described in the user manual in the "Maintenance and care" chapter may be performed by the operator.

- Only clean the exterior of the device with a slightly moistened, non-dripping cloth. Use only water and, if required, customary surfactants.
- The operator is responsible for establishing appropriate safety precautions for cleaning the sample compartment and transport channels (hose system) of the device. This applies in particular to contaminated and infectious materials.
- Use only original spare parts, wear parts and consumables. They have been tested and ensure safe operation. Glass parts are wear parts and are not subject to the warranty.

2.6 Behavior during emergencies

Observe the following:

- If there is no immediate danger of injury, extinguish the plasma immediately via the plasma deactivation switch in the event of danger or accidents.
- If possible, switch off the device via the power switch after a cooling down period of 30 s. Then disconnect the mains plugs of the device and the system components from their power sockets.
- Close the gas supply as soon as possible after switching off the devices.

3 Function and design

3.1 Function and measuring principle

ICP emission spectrometry (ICP-OES) uses of plasma at temperatures of up to 10000 K. This high temperature is focused in a very small area of approx. 5 cm³. The sample is introduced to this plasma in aerosol form (small droplets in a gas). The droplets dry, melt, vaporize and are atomized or ionized. During this process, the analysis channel of the plasma through which the sample is flowing will cool down to approx. 6000 to 7000 K. Atoms and ions are excited to emit light at these high temperatures. The light is broken down by the device optics into wavelengths ("colors") whose intensity is measured to indicate concentrations. A detector is used to measure the intensity of the emission line and its spectral environment. The net intensity of the measured signal is used as the measurand ("peak").

The inert gas argon is used as operating gas. This gas flows inside a plasma torch made up of three concentric pipes. The plasma gas (also called cooling gas) flows at a rate of approx. 10 to 18 l/min on the outside to cool the external torch pipe. The sample aerosol is injected in the plasma in the innermost pipe, hence its name "injector". The sample aerosol is created shortly before with a nebulizer and a downstream spray chamber in which larger droplets are separated.

The exhaust heat of the plasma is dissipated partly by the recirculating chiller and partly by the exhaust unit.

3.2 Design

Essentially, the optical emission spectrometer consists of the following components:

- Components for plasma generation (HF generator, induction coil, torch)
- Sample supply system with hose pump, nebulizer and spray chamber
- Optical system with transfer optics, spectral photometer and detector

Both models, the PlasmaQuant 9100 and the PlasmaQuant 9100 Elite differ with regard to their optical systems. The plasma generation and sample supply systems are identical. The more powerful model, the PlasmaQuant 9100 Elite with high-resolution optics, is particularly suitable for interference-free analysis of samples in complex matrices. Important application fields are the analysis of rare earths, of high-alloy steel or petrochemical product. The standard model, the PlasmaQuant 9100, achieves very good results in routine analysis despite its slightly low resolution.

Sample chamber and plasma compartment

The sample introduction system is freely accessible in the sample chamber. The torch and the induction coil, however, are located in the shielded plasma compartment to protect the user from the high-frequency radiation and the UV radiation from the plasma. The spatial separation of sample supply and plasma also prevents the heat from the plasma being transferred to the spray chamber without obstruction and causing a drift there.



Figure 1 Emission spectrometer, with opened plasma compartment

3.2.1 Plasma generation

HF generator

The emission spectrometer uses a free-running HF generator (high-frequency generator) with a frequency of 40.68 MHz. With the help of the high performance coil, the HF generator has an output of 700 to 1700 W into the plasma. The fully automatic adjustment of the power relative to the actual current sample load in the plasma provides high plasma consistency. This makes the plasma very robust and able to manage difficult sample matrices such as organic solutions or salt loads. The HF generator is located immediately behind the plasma compartment and is shielded separately because of the high intensity of high frequencies.

The power for maintaining the plasma is transferred to the torch in the plasma compartment by means of an induction coil with four windings. The induction coil is water-cooled.

During the initial plasma ignition period, a high voltage spark is passed from the ignition spark generator to the discharge spring in the high-frequency field in the torch. The discharge spring is located near the induction coil.

Torch

The torch is designed with three layers and consists of an outer tube, an inner tube and the injector tube on the inside.

The outer tube, in conjunction with the bonnet inserted into the coil, electrically insulates the plasma from the induction coil and shields the plasma from the ambient air. The plasma gas flows between the outer and inner tube. This gas is ionized at the induction coil and converted to a plasma state. The tangential flow of the plasma gas into the space between the outer and inner tube cools the outer tube and prevents it from melting due to the high plasma temperatures.

The auxiliary gas flows in the intermediate space between the inner tube and the injector. It pushes the aerosol of the sample solution away from the injector.

The injector has the task of injecting the sample aerosol into the plasma. The measurement solution is transported by the nebulizer gas from the spray chamber through the injector into the plasma.

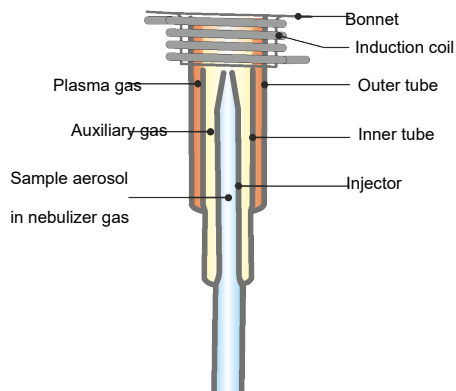


Figure 2 Torch diagram with gas flows

The torch can be disassembled into its individual parts ("demountable torch"). Worn individual parts can be replaced individually, such as the outer tube, which is subject to high thermal loads. Care must be taken during assembly to ensure that the individual parts are inserted gas-tight and especially to check the proper fit of the injector. The standard torch (made of quartz glass with injector, 2 mm) is also available as a unit. The one-piece torch is cleaned as a whole; disassembly and subsequent assembly are omitted. On the other hand, this torch has to be completely replaced when worn.

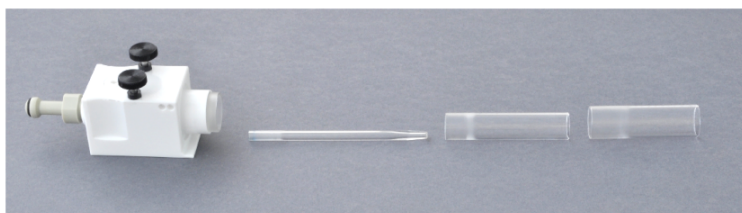


Figure 3 Demountable torch



Figure 4 One-piece torch

A special design (V Shuttle Torch) is used in the emission spectrometer. The upright positioning of the torch reduces clogging and soot formation.

The torch is mounted onto the rail guide with the holder (shuttle). This automatically connects the internal gas inlets for the plasma and the auxiliary gas. The torch is then moved by hand along the rail guide to the plasma compartment where it locks in the adjusted operating position.

3.2.2 Sample supply

Pump, nebulizer and spray chamber

A hose pump evenly conveys the measurement solution to the nebulizer. The rotational speed of the pump and the diameter of the pump tubing used determine the amount of sample delivered. By using the pump, sample introduction into the nebulizer, and thus also the sensitivity of the measurement signal, is independent of the viscosity of the measurement solution.

The aerosol of the measurement solution required for atomization/ionization in the plasma is generated by a pneumatic concentric nebulizer. An argon flow is directed past the sample nozzle of the nebulizer as nebulizer gas. The gas flow continuously tears the surface of the liquid at the nozzle and produces small sample droplets. The sample aerosol that is formed is transported with the nebulizer gas through the spray chamber to the plasma. A cyclone chamber is used as the spray chamber. On the way through the spray chamber, large droplets in the spray chamber are separated by the centrifugal force and flow out via the waste outlet.



Figure 5 Concentric nebulizer and spray chamber

An ultrasound nebulizer is provided as an optional accessory. It delivers a high aerosol yield for aqueous solutions. Also, the ultrasound nebulizer can remove interfering solvents from the measuring gas via control of the temperature (heating range: 120 to 160 °C, cooling range: -20 to +10 °C). This results in high signal intensity. It increases the sensitivity and lowers the detection limits.

Special sample supply systems

Analytik Jena GmbH provides optimized sample supply systems for special applications.

Sample supply system	Application
Standard Kit	Standard applications: Environmental samples, foodstuffs, pharmaceuticals
HF Kit	Digestions containing hydrofluoric acid: Metals, ceramics, rare earths
Organic Kit	Organic samples: Crude oil, petrochemical products such as kerosene, organic solvents
Salt Kit	Highly saline sample: Brine, corrosive solutions or solutions containing sulfur, seawater

3.2.3 Optical system

In both device models, analyte emission in plasma is observed from two directions, axial and radial (DualView PLUS). The emission radiation is selectively coupled via the transfer optics from either direction to the monochromator. The working range is increased by attenuating both observational directions.

For the PlasmaQuant 9100 Elite, the selectivity of analysis is implemented by the high-resolution double monochromator on the basis of a prism and an echelle grating monochromator (high-resolution optics). Due to the echelle grating's large blaze angle of 76°, spectral resolution is 0.002 nm at 200 nm.

The spectral resolution of the PlasmaQuant 9100 is 0.006 nm at 200 nm.

On both models, the monochromator is wavelength-stabilized via the use of an integrated neon emitter. Wavelength reproducibility is achieved by the internal Ne-line calibration of the monochromator when approaching a wavelength.

A low noise, UV-sensitive semiconductor detector (CCD line detector) is located at the exit slit of the monochromator. This detector not only registers the intensity of the analysis line, but also its spectral neighborhood. In this way, a spectral range of approx. 1 nm in the vicinity of the analysis line is detected simultaneously and at a high resolution.

3.3 Connections

3.3.1 Supply and control connections

The supply lines of the emission spectrometer are connected by the service engineers during installation.

The green power switch and the red plasma deactivation switch are located on the left-hand side of the device. Also on the left side, under a cover plate, are the connections for PC and accessories as well as the media connections for gas and the cooling water inlet and outlet.

A pair of carrying handles are fastened to the left and right for transport and installation. The handles are unscrewed and removed after installation. The carrying handles must be stored in a safe place in case the device has to be transported again or moved within the laboratory.



Figure 6 Connections on the left side of the device

- | | |
|-------------------------------------|---------------------------------|
| 1 Manual plasma deactivation switch | 2 Mains switch |
| 3 Opening for carrying handle | 4 Connections for cooling water |
| 5 Gas connectors | 6 Water filter |
| 7 Power cable | 8 Fuses |
| 9 Interfaces | 10 Type plate |

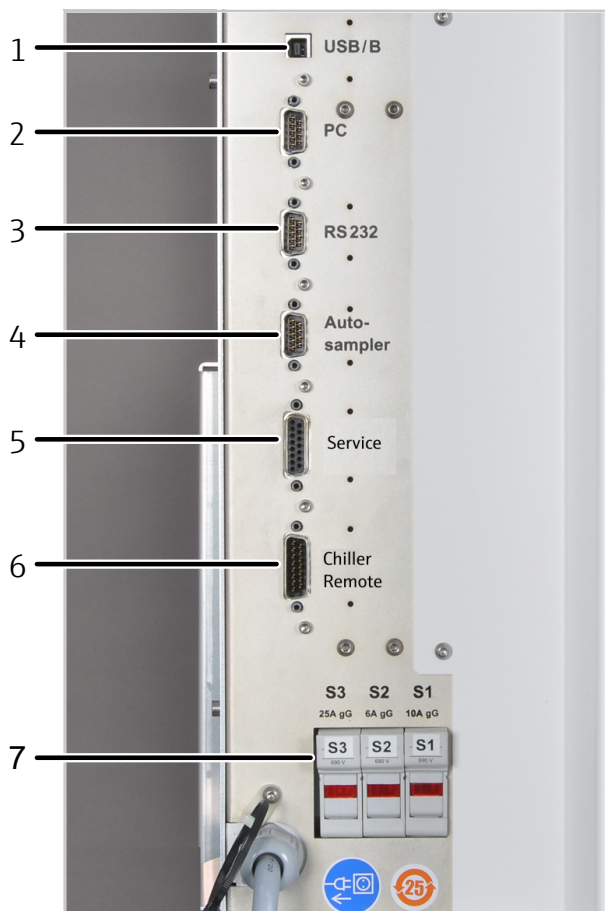


Figure 7 Interfaces and fuses

- 1 "USB/B" for PC USB connection
- 2 "PC" for serial PC connection (optional)
- 3 "RS 232" (service only)
- 4 "Autosampler" for sampler serial connection (12 V)
- 5 "Service"
- 6 "Chiller Remote"
- 7 Instrument fuses S1, S2, S3

The "Chiller Remote" connection optionally allows the recirculating chiller to be controlled via the emission spectrometer.

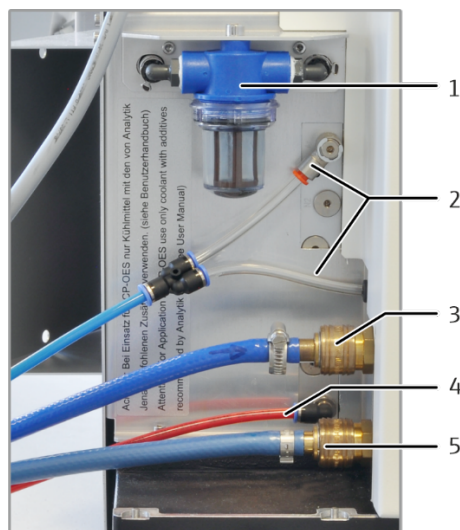


Figure 8 Connections for gases and cooling water

- | | |
|-----------------------------------|---|
| 1 Water filter in cooling circuit | 2 Connections for argon |
| 3 Cooling water inlet "IN" | 4 Connection for oxygen as an additive gas (optional) |
| 5 Cooling water outlet "OUT" | |

In the device, argon is used as a gas for the torch, the nebulizer and for purging the spectrometer. The purge gas is then directed via the cone for axial observation as a counter gas to avoid a heavy load being placed on the cone and plasma window by the plasma torch. Oxygen can be optionally connected as an additive gas.

Plug-in connectors are used for the gas connections of the device. Hoses are pushed into the connector as far as possible and are thus securely fastened. To release the connection, you have to push the colored ring inwards and pull the hose out at the same time.

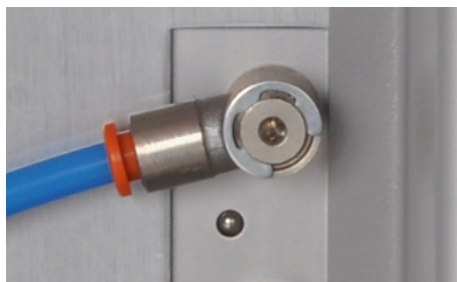


Figure 9 Plug-in connectors for gas

The hoses for cooling water are also equipped with quick couplings. When connecting the hoses, the connecting pieces on the hoses are pushed into the sockets all the way until they engage with a click. When releasing the connection, you have to push the ring on the socket back and pull the hose out of the connection. The valves in the quick couplings prevent the cooling water from flowing out.

Type plates are attached to the top of the terminal strip and behind the door of the plasma compartment. The type plate contains the following information:

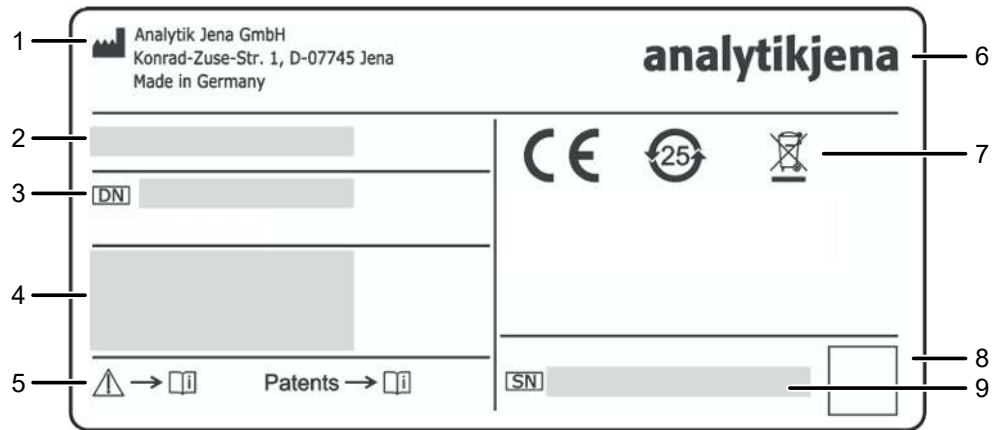


Figure 10 Type plate

- | | |
|---|------------------------------|
| 1 Manufacturer address | 2 Trade name |
| 3 Device number | 4 Electrical connection data |
| 5 Follow instructions. Observe patents (optional) | 6 Trademarks |
| 7 Symbols: CE mark, WEEE label, etc. | 8 QR code |
| 9 Serial number | |



Figure 11 Rear of the device

- | | |
|--------------|----------------|
| 1 Air filter | 2 Exhaust vent |
| 3 Fan | |

3.3.2 Connections in the plasma compartment and in the sample chamber

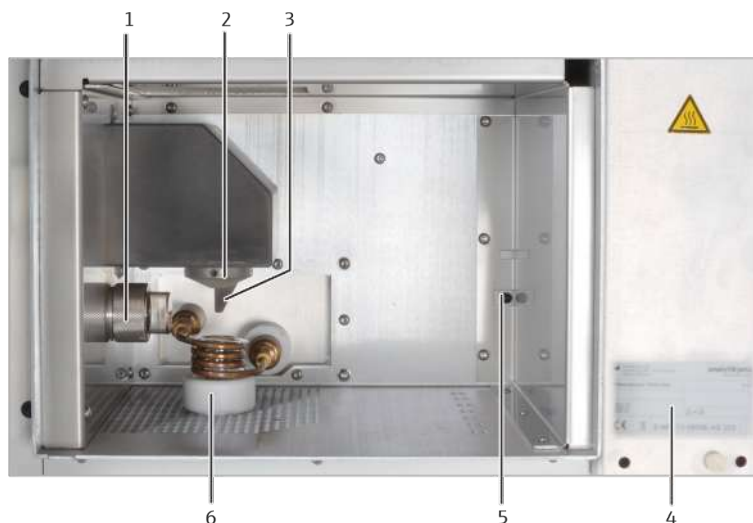


Figure 12 Plasma compartment

- | | |
|---------------------------------------|-------------------------------------|
| 1 Window for radial observation | 2 Cone for axial observation |
| 3 High-voltage (HV) discharge spring | 4 Type plate |
| 5 Photodetector for plasma monitoring | 6 Induction coil with bonnet, torch |

The serial number of the HF generator is located in the plasma compartment.



Figure 13 Sample chamber

- | | |
|-------------------------------|-------------------------------------|
| 1 Torch | 2 Mechanical height adjustment |
| 3 Fork clamp | 4 Nebulizer with sample intake tube |
| 5 Hose pump | 6 Drip tray |
| 7 Argon hose on the nebulizer | 8 Waste hose on the spray chamber |
| 9 Argon hose | 10 Spray chamber |

3.4 ASPQ 3300 sampler

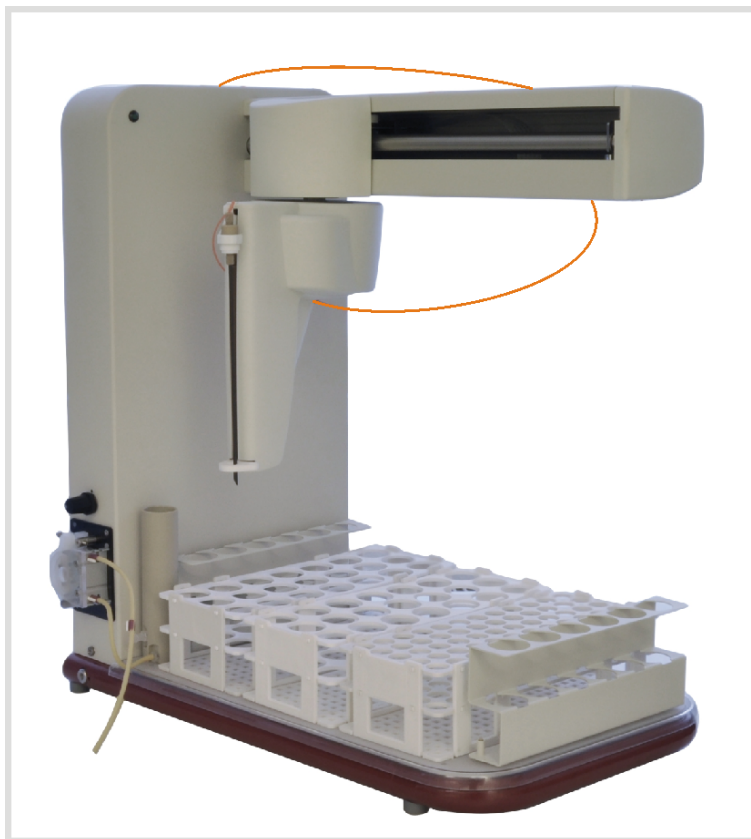


Figure 14 ASPQ 3300 sampler

The sampler facilitates fully automatic routine analysis. It can be equipped with 3 sample racks and 2 racks with 6 special samples, e.g. standard solutions.

The following sample racks are available:

Rack/number of samples	Vessel volume
6 (special vessels)	50 ml
21	50 ml
24	30 ml
40	20 ml
60	14 ml
7 ml	7 ml

The purging vessel is installed on the sampler. The hose pump at the sampler pumps the purging liquid from the storage bottle into the purging vessel – this action cleans the inserted cannula by cleansing it inside and out. Excess purging liquid is pumped into the waste container during purging. The purging liquid for between measuring breaks or for special purging steps within a measuring routine is also taken from the purging vessel.

A power connection supplies operating voltage to the sampler.

3.5 Other accessories

Hg/hydride systems	<p>Two Hg/hydride systems are available for determining mercury and hydride-forming metals:</p> <ul style="list-style-type: none">■ HS Pro PQ – for targeted determination of Hg/hydrides with highest detection strength■ HS PQ – for simultaneous determination of Hg/hydrides with the classic elements
Sampler	<ul style="list-style-type: none">■ Teledyne Cetac ASX-560 sampler■ Cetac Oils 7400 sampler <p>The Teledyne Cetac ASX-560 sampler is suitable for aqueous solutions and has an integrated purging function. It can be equipped with different sample racks and an extra rack for standard solutions.</p> <p>The Cetac Oils 7400 sampler allows automatic supply of oils and coolants. It includes a stirring function and a double purging station for operation with various sample types. The sampler also has a drip catcher to prevent cross-contamination.</p> <p>Both samplers can be coupled with the Cetac ASXPress Plus switching valve.</p>
Dilution system	<ul style="list-style-type: none">■ Teledyne Cetac SDX(HPLD) dilution system <p>The dilution system can dilute samples up to 1:5000. The integrated vortex mixer mixes the samples with the diluent. The dilution system can dilute responsively and intelligently. The dilution system is controlled via the ASpect PQ software. The operator can set parameters such as the maximum dilution factor or the vortex speed easily in the software.</p> <p>The dilution system is coupled with the Teledyne Cetac ASX-560 sampler.</p>
Accessories for quick sample supply	<ul style="list-style-type: none">■ Cetac ASXPress Plus aqueous accessory■ Cetac ASXPress Plus oil accessory <p>Both accessories for aqueous samples or oils shorten sample intake time and also the purging times. Higher sample throughput can be achieved. The accessories consist of a switching valve with a vacuum pump, and are supplied with their own control unit.</p>
Temperature-controlled spray chamber	<p>The IsoMist XR temperature-controlled spray chamber has an integrated Peltier element to bring the spray chamber to temperatures of -25 °C to +80 °C (in steps of 1 °C).</p> <p>The spray chamber is particularly suitable for organic analyses. It increases the temperature stability of the sample supply system. Cooling of the samples leads to decreased amounts of solvent vapor in the spray chamber.</p> <p>The temperature of the spray chamber is controlled via its own software included with the accessory. Data is transmitted between the spray chamber and the PC optionally via USB cable or wireless technology (Bluetooth).</p>
Argon humidifier with bypass	<p>The argon humidifier is suitable for analysis of samples with high salt content. By humidifying to nebulizer gas, the argon humidifier prevents salts from crystallizing in the nebulizer or injector and blocking it. The argon humidifier also improves signal stability and recovery.</p> <p>Argon flows over a membrane coil through deionized water and is saturated with water vapor. Argon humidification can be switched on and off simply with the aid of the bypass valve without disconnecting any hoses.</p>
Inline filter	<p>The inline filter is suitable for analysis of samples with high solids content. The inline filter prevents solids from being deposited in the nebulizer or injector and blocking it. It also improves signal stability and recovery.</p>

Accessories description

Descriptions of the accessories can be found in the corresponding accessory user manual. This user manual only describes the installation of a temperature-controlled spray chamber, argon humidifier and inline filter.

The operator is also instructed in coupling the Teledyne Cetac ASX-560 sampler with the Teledyne Cetac SDX(HPLD) dilution system and Cetac ASXPress Plus switch valve.

4 Installation and commissioning

4.1 Installation conditions

4.1.1 Installation location requirements

The emission spectrometer may only be operated in closed rooms. The location must have the quality of a chemical laboratory (indoor use).

- Avoid direct sunlight and radiation from heaters on the device. Air conditioning is recommended at the location. The cool air emanating from the air conditioning unit should not be directed at the device.
- Do not place the device near sources of electromagnetic interference.
- A separate room is recommended for sample preparation and storing liquid chemicals.

The climate conditions required for the room the device is operated in are:

Temperature range	+15 °C to +35 °C, optimal is +20 °C to +25 °C, as constant as possible during measuring operations
Max. humidity	20 to 90 % at 20 °C
Air pressure	0.7 bar to 1.06 bar
Max. permissible altitude	2000 m
Storage	Temperature: -40 °C to +70 °C Use dessicant

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.

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

Optimum device function strongly depends on a correct grid connection with an adequate cable cross-section. The power connection must be protected on the building side with a 32 A slow-blow fuse and must be installed prior to delivery of the device near the installation location. The length of the device cable is 3 m. The CEE surface box (2-pole + E Blue 5UR 3 206-2 220/32) is provided as per the supply contract.

To prevent sudden voltage spikes or drops, do not connect the device to electrical circuits also connected to other power-intensive devices.

Voltage	230 V \pm 10%
Frequency	50/60 Hz
Typical average power consumption	4500 VA
Maximum current consumption	32 A
Fuse protection (grid side)	32 A

4.1.3 Gas supply

The following gases are used with the emission spectrometer:

- Argon as the gas for the torch (plasma gas, auxiliary gas, nebulizer gas)
- Argon as the purging gas for the spectrometer and as the cone gas
- Oxygen as an auxiliary gas

Optionally for selected applications, such as with some organic solvents, oxygen can be used as an addition to the nebulizer gas.

The standard length of the hose is 3 m. If different hose lengths are required, please contact customer service.

Gas	Inlet pressure	Total consumption
Argon \geq 4.6	600 kPa (6 bar)	13 to 21 l/min
Permissible components: Oxygen \leq 3 ppm Nitrogen \leq 10 ppm Hydrocarbons \leq 0.5 ppm Moisture \leq 5 ppm		
Oxygen \geq 4.5 (as optional auxiliary gas)	600 kPa (6 bar)	\leq 0.04 l/min

4.1.4 Exhaust unit

The exhaust unit must be switched on during operation of the emission spectrometer. Whether the exhaust is switched on is checked via the device-internal safety circuits before plasma ignition. The plasma will not be ignited if a fault is present.

Correct exhaust ventilation requires sealed connection of a suction hose to the smoke-stack of the emission spectrometer.

The exhaust unit is to dissipate health-hazardous gases created during plasma operation such as ozone or nitrous oxides. Use an exhaust unit made of heat- and corrosion-resistant material. The first 6 m of the exhaust system must be made of metal or a heat-resistant material (> 85 °C). The first meter must be made of a flexible material to reach the device from above.

Material	Heat and corrosion resistant (recommended: V2A steel)
External pipe diameter	125 mm
Exhaust performance	3.5 m ³ /min (min.), 5.5 m ³ /min (max.) Optimum: 4.0 to 4.5 m ³ /min
Adapter using flexible aluminum pipe	Pipe diameter: 125 mm Pipe length: 1000 mm

4.1.5 Recirculating chiller

The HF generator is cooled by the cooling circuit of the external recirculating chiller. Please observe the information provided in the recirculating chiller's operating instructions.

The recirculating chillers supplied by Analytik Jena GmbH are adapted to the required cooling performance of the emission spectrometer.

If the recirculating chiller is not obtained from Analytik Jena GmbH, the following requirements must be met:

Water flow in the cooling water circuit	1.5 to 2.0 l/min
Cooling water temperature range at device cooling water inlet	17 to 24 °C
Cooler target temperature	18 °C
Temperature stability	± 0.1 °C
Conductivity of the cooling water	50 to 200 µS/cm
Cooling performance	3000 VA
Pressure setting (max.)	600 kPa (6 bar)



NOTICE

Danger of corrosion in the cooling water circuit

In addition to the risk of corrosion, base metals increase the conductivity of the cooling water.

- When selecting the recirculating chiller, make sure that no base metals are used in components carrying water.

The recirculating chiller must be filled with cooling water that has been mixed with the cooling water additive from Analytik Jena GmbH (→ "Maintenance of the recirculating chiller: Replacing the cooling water" ☰ 76). The coolant additive prevents potential damage to the emission spectrometer resulting from corrosion and biological contamination. Damage to the device due to operation without coolant additive is excluded from the warranty!

For night-time and constant operation the recirculating chiller can be controlled via the emission spectrometer. Analytik Jena GmbH supplies a matching communication cable with the recirculating chiller. The cable connects the "Chiller remote" connection on the left-hand side of the emission spectrometer to the interface on the right-hand side of the chiller (→ "Supply and control connections" ☰ 17). The chiller is then switched on and off automatically as the plasma ignites and goes out.

4.1.6 Device layout and space requirements

The emission spectrometer is a compact device designed for table-top operation. The required space depends on all the components that make up the measuring station.

Components of the measuring station:

- Autosampler
- Recirculating chiller
- Waste bottle (under the bench)
- The PC and printer may be placed on a separate side table.

The workbench must meet the following requirements:

- The dimensions of the workbench for the device and autosampler must be at least 1800 mm x 750 mm. A minimum distance of 300 mm must be maintained between the rear of the device and the closest wall.
- The height can be chosen according to ergonomic aspects.
- The device must be freely accessible from all sides.
- The workbench must be capable of bearing a load of at least 200 kg.
- The workbench surface must be wipe, scrape and corrosion resistant and must not absorb moisture.

Component	Width x height x depth [mm]	Mass [kg]
On the workbench		
Basic device	990 mm x 940 mm x 855 mm	170 kg
ASPQ 3300 sampler	285 mm x 510 mm x 490 mm	15 kg
Teledyne Cetac ASX-560 sampler	580 mm x 620 mm x 550 mm	12 kg
Cetac Oils 7400 sampler	570 mm x 490 mm x 540 mm	23 kg
Teledyne Cetac SDX(HPLD) dilution system	132 mm x 254 mm x 117 mm	4.4 kg
Switch valve Cetac ASXPress Plus with control unit	58 mm x 128 mm x 217 mm 83 mm x 254 mm x 200 mm	1.3 kg 1.4 kg
Outside the laboratory or next to the workbench		
Water-air cooler	460 mm x 703 mm x 735 mm	92 kg
Water-water cooler	360 mm x 590 mm x 470 mm	33 kg (empty)
Under the workbench		
Waste bottle (∅ x height)	120 mm x 250 mm	

To ensure unimpeded intake and exhaust of cooling air and effective cooling, the surfaces of the housing sides of the water-air cooler require a minimum distance of 60 cm from adjacent objects.

Due to the waste heat generated and the possible noise pollution, it is recommended to place the water-air cooler outside the laboratory. An extension of the cooling water hoses is permitted if minimum pressure and flow volume are maintained. The cooler must be located on the same floor as the basic device. If this is not the case, additional check valves must be installed in the water circuit. Otherwise the water tank can run empty when the device is at a standstill. These adjustments will not be carried out by Analytik Jena.

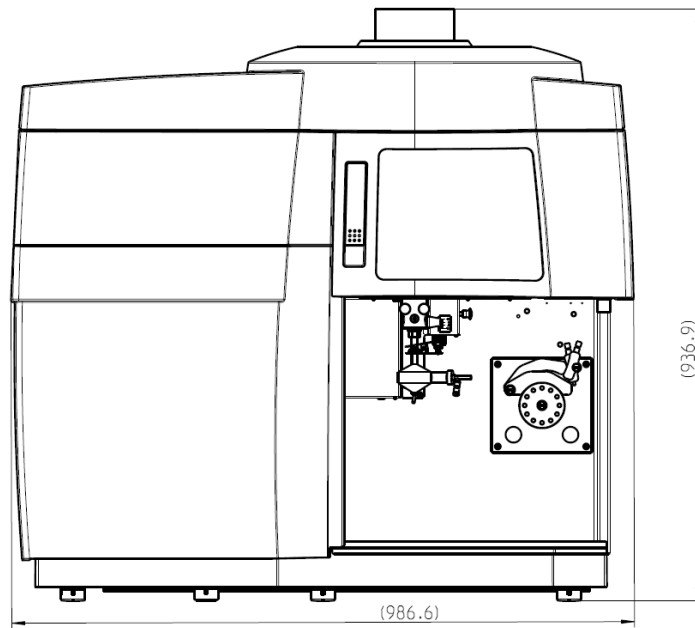


Figure 15 Space requirements (viewed from the front)

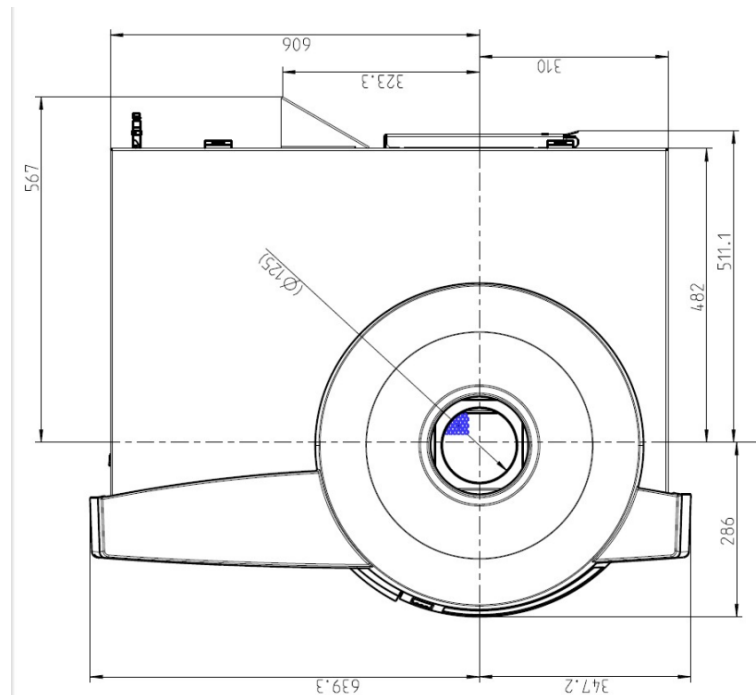


Figure 16 Space requirements (view from above)

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 GmbH or by persons authorized by Analytik Jena GmbH.

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 sample supply system

The systems for sample supply, the torch and the nebulizer with spray chamber and the sampler must be installed by the customer during maintenance.



CAUTION

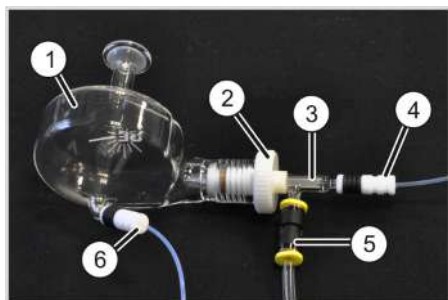
Risk of injury

A risk of injury due to broken glass is present when handling glass parts.

- Handle glass parts with extreme caution.



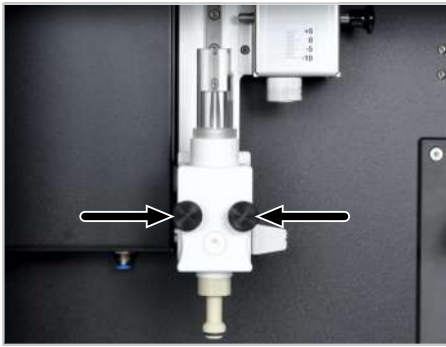
- ▶ Place the bonnet in the induction coil. The beveled edge of the bonnet must rest on the topmost coil winding.



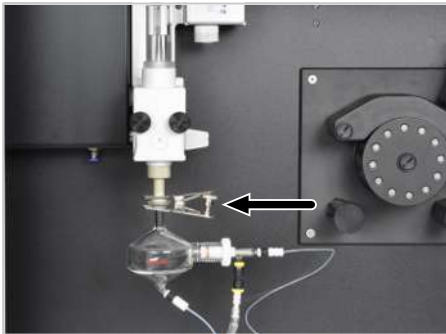
- ▶ Attach the sample hose (4) and the carrier gas hose (5) to the nebulizer.
- ▶ Loosen the plastic nut (2) on the spray chamber (1). Push the nebulizer (3) into the spray chamber as far as possible and tighten the plastic nut hand-tight.
The connector to connect the carrier gas to the nebulizer should point downward.
- ▶ Attach the waste hose (6) to the bottom connector of the spray chamber.



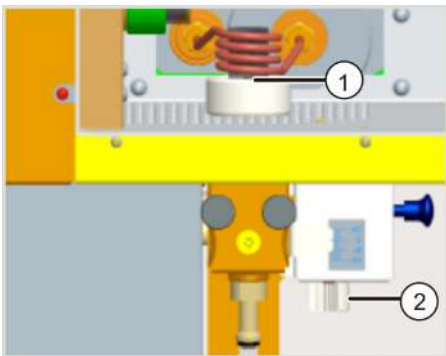
- ▶ Check on the carriage if the O-rings are in the gas connections. (The O-rings can stick to the torch holder when the torch is removed).



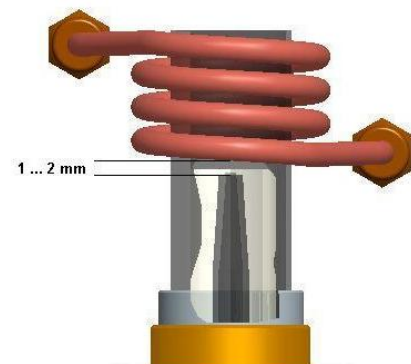
- ▶ Attach the torch to the carriage on the adjusting rail in the sample chamber and screw it on.
NOTICE! Tighten the screws firmly so that the gas supply is leak-proof.



- ▶ Fit together the spherical joint connection of the torch and the spray chamber and secure the connection with the fork clamp.
- ▶ Push the torch upwards on the adjusting rail until it engages with the height adjustment.



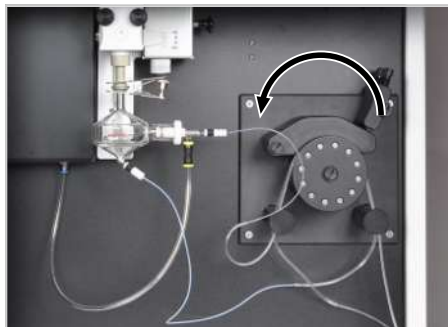
- ▶ Adjust the torch with the manual height adjustment (2) so that the tip of the injector in the torch (1) is situated approx. 1 – 2 mm below the lowest winding of the induction coil.



- ▶ The outer tube must be removed to adjust the ceramic torch (HF kit).
 - Adjust the torch with the manual height adjustment (2) so that the outer edge of the inner tube is situated approx. 1 – 2 mm below the lowest winding of the induction coil.
 - Reinsert the outer tube after adjustment.



- ▶ Cut the sample and waste hoses to the required length, ensuring they are long enough that the torch with the nebulizer unit can still move freely on the adjusting rail. Slightly chamfer the ends of the hoses.
- ▶ Connect the sample hose of the nebulizer to the pump hose with the black stoppers and the waste hose to the pump hose with the red stoppers. Push the ends of the sample and waste hoses several millimeters into the pump hoses.
To improve grip on the hoses, use a piece of fine sanding paper as a grip.



- ▶ Clamp each of the pump hoses into the pump between two stoppers. **NOTICE!** Note the pump flow direction! The pump rotates in a counter-clockwise direction!
- ▶ Place the clamping brackets over the hoses. Make sure the pump hoses are in the grooves of the clamping brackets. Fasten the clamping brackets via the clamping levers; the clamping levers must engage audibly.
- ▶ Connect the sample pump hose to the hose of the sampler (for automatic operation) or to a hose leading directly into the sample (manual operation).
- ▶ Connect the waste hose on the waste pump hose to the waste container. **NOTICE!** The waste hose must not dip into the liquid! This prevents waste material from being pumped into the nebulizer system in case of faulty pump hose connection.

Notes on the pump hoses

Different materials can be selected for the pump hoses depending on the sample. The inner diameter of the waste hose is one and a half times the size of that of the sample pump hose. This ensures that sample solution separated from the aerosol is transported away quickly and the spray chamber does not fill up.

Pump hose	Inside diameter	Identification (stopper)
Sample supply	0.762 mm/0.03 inch	B/black
Waste	1.143 mm/0.045 inch	Red/red

Set the contact pressure on the pump hose as follows:

- ▶ Loosen the screw of the contact pressure level so that no liquid is transported.
- ▶ Slowly tighten the screw until liquid begins to flow along the hose.
- ▶ Tighten the screw by a further half-turn.

If the pump is out of operation, release the clamping bracket. This extends the service lives of the pump hoses.

4.3 Putting the ASPQ 3300 into operation

Connections

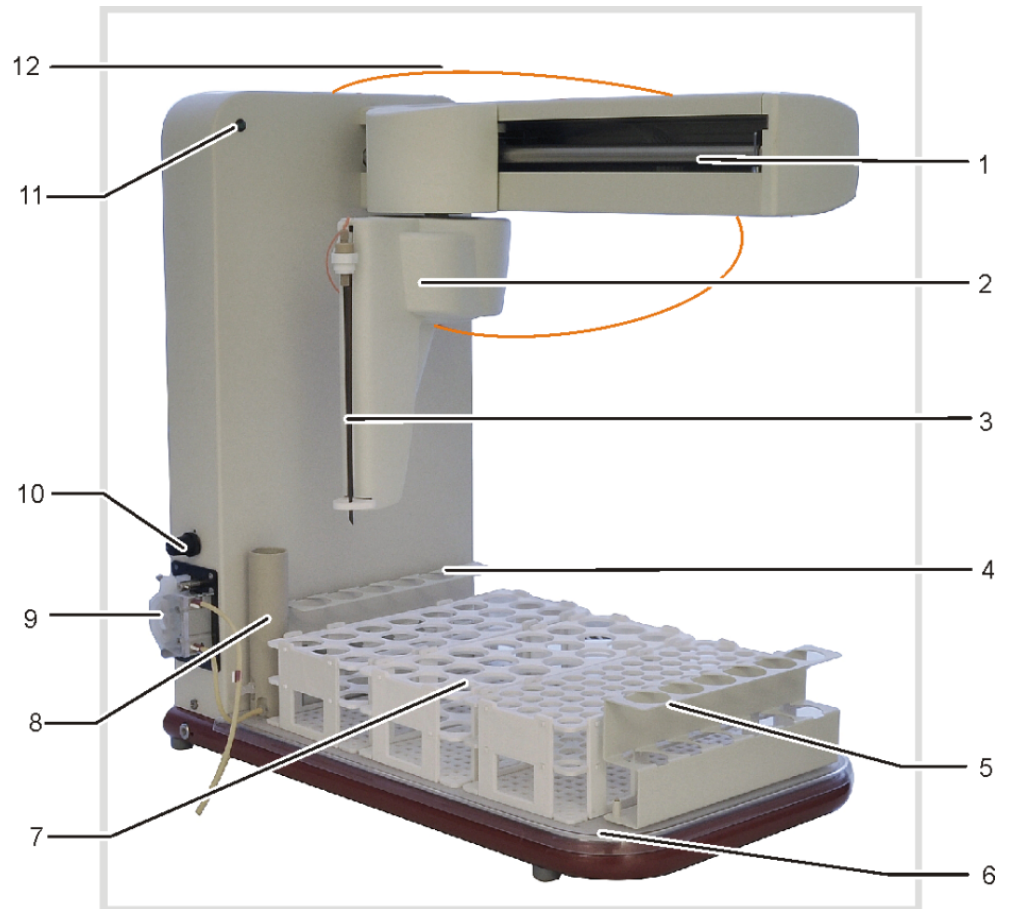


Figure 17 ASPQ 3300 sampler

- | | |
|----------------------------|---------------------------------------|
| 1 Sampler arm | 2 Sampler head with cannula holder |
| 3 Cannula | 4 Rack for special samples |
| 5 Rack for special samples | 6 Base plate for racks |
| 7 Sample racks | 8 Purging vessel |
| 9 Purging vessel pump | 10 Controller for purging vessel pump |
| 11 Power LED | 12 Sample intake hose |

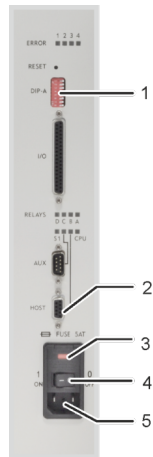


Figure 18 Connection panel on the right side of the sampler

- | | |
|--------------------|---|
| 1 DIP switch | 2 "HOST" connection (to the basic device) |
| 3 Power switch | 4 Fuse holder |
| 5 Power connection | |

Note: DIP switch 5 is set to "ON".

Only the aforementioned connections are required for the use of the sampler together with the basic device. All other connections and displays are for service purposes or not in use.

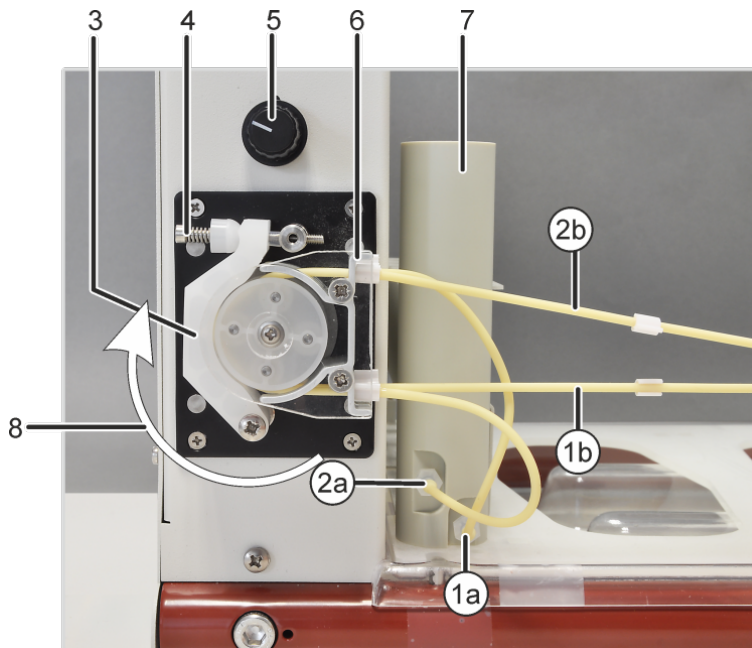


Figure 19 Purging vessel and pump on the sampler

- | | |
|---|--|
| 1a Intake connection for purging solution on the purging vessel | 1b Purging solution hose |
| 2a Waste connection on the purging vessel | 2b Hose to the waste container |
| 3 Clamping bracket | 4 Clamping lever with spring |
| 5 Pump speed controller | 6 Hose block for clamping the pump hoses |
| 7 Purging vessel | 8 Pump direction |

Installing the sampler



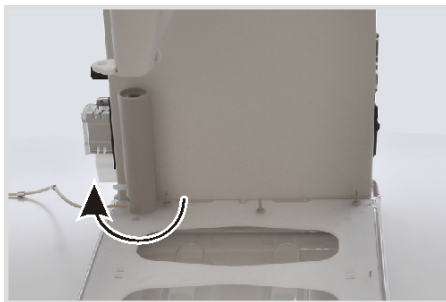
NOTICE

Risk of damage to the sensitive electronics

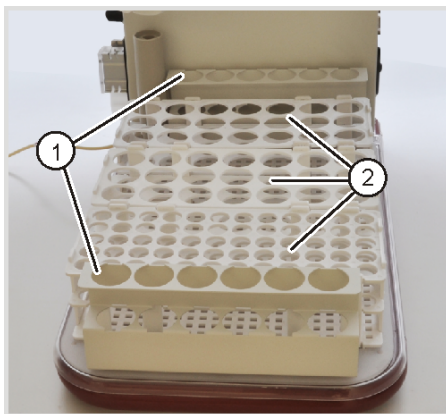
- Only connect the sampler to the electrical grid after installation.



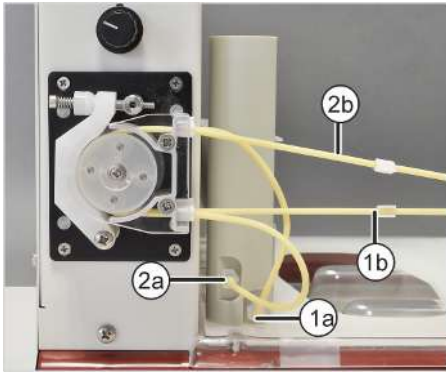
- ▶ Place the tray on the automatic sampler foot, and place the base plate for holding the sample racks on that. In this case, the place for the purging vessel must be located on the left to the rear. The base plate is correctly mounted if it fails to move after light shaking.



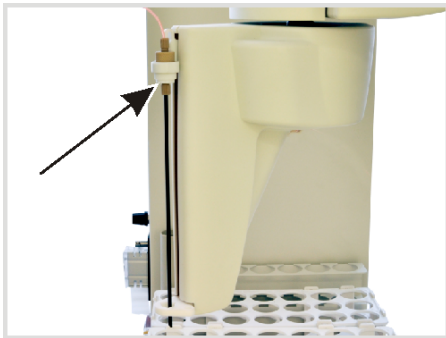
- ▶ Installing the purge vessel: Insert the purging vessel into the rear left recess and turn it clockwise by 90°.



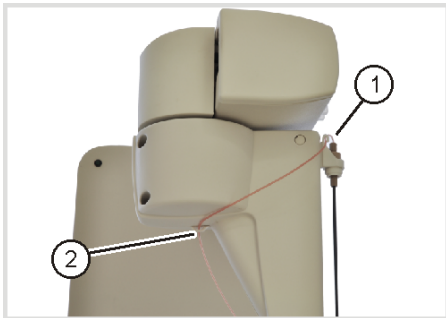
- ▶ Fit the racks for special samples (1) on the base plate and attach the required sample racks (2). The sample positions are coded with a three-digit number (e.g. 108) in the control software. The first number indicates the sample rack itself, the following numbers the position on the rack. The first sample rack is located in front of the purging vessel, followed by the second and third. In the software the positions are only shown for illustration.



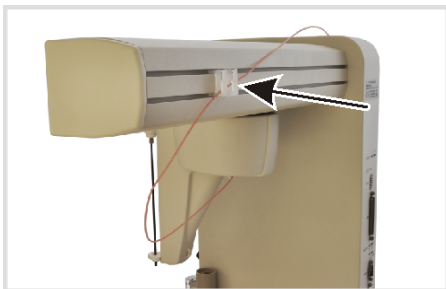
- ▶ Connect the pump hose for the purging solution to the lower intake connection (1a) of the purging vessel. Place the pump hose over the hose block from above and clamp it between two stoppers. Connect the intake hose for purging solution to the other end of the hose (1b). Immerse the intake hose in purging solution.
- ▶ Connect the pump hose for waste to the top outlet connection (2a) of the purging vessel. Place the pump hose over the hose block from below and clamp it between two stoppers. Connect the waste hose to the other end of the hose (2b). Insert the waste hose in the waste bottle.
NOTICE! Note the pump direction! The pump moves in a clockwise direction.
- ▶ Fasten the clamping bracket over the pump hoses via the clamping lever.



- ▶ Insert the cannula into the holder on the sampler head.
 - Move the holder along the Z-axis (down and up) and check whether the cannula fits through the guide at the lower end of the head.
 - Fasten the cannula on the holder with the nut (arrow in figure on the left).



- ▶ Initially guide the sample hose in an arc through the eyelet on the cannula holder (1).
- ▶ Thread the hose from the left side through the eyelet (2) on the lower end of the head.



- ▶ Place the hose to the rear in the eyelets on the rear of the sampler arm.
- ▶ Connect the hose with the sample hose of the basic device.



- ▶ Check the DIP switch (1). Set switch 5 to "ON"; all other switches remain at their initial positions.
- ▶ Connect a USB cable to the "Host" connection and to the "Autosampler" connection on the basic device.
- ▶ Connect the power supply cable to the power connection (3) and to a grounded power outlet.
- ▶ Set the pump speed on the sampler so the liquid level remains constant and not too much purging liquid overflows.

4.4 Installing other accessories

4.4.1 Coupling the Teledyne Cetac ASX-560 sampler with other accessories

The following instructions describe connecting the Teledyne Cetac ASX-560 sampler with the Teledyne Cetac SDX(HPLD) dilution system and the Cetac ASXPress Plus switching valve, and connection to the emission spectrometer.

When supplied together with the emission spectrometer, the accessories are commissioned together with the basic device. The operator is only required to perform installation if the accessories were ordered and supplied individually.

Details descriptions for installing the accessories can be found in the corresponding user manuals.

Connecting the sampler and the dilution system

- ▶ Connect the sampler and the dilution system via the following interfaces and connect them to the power grid:

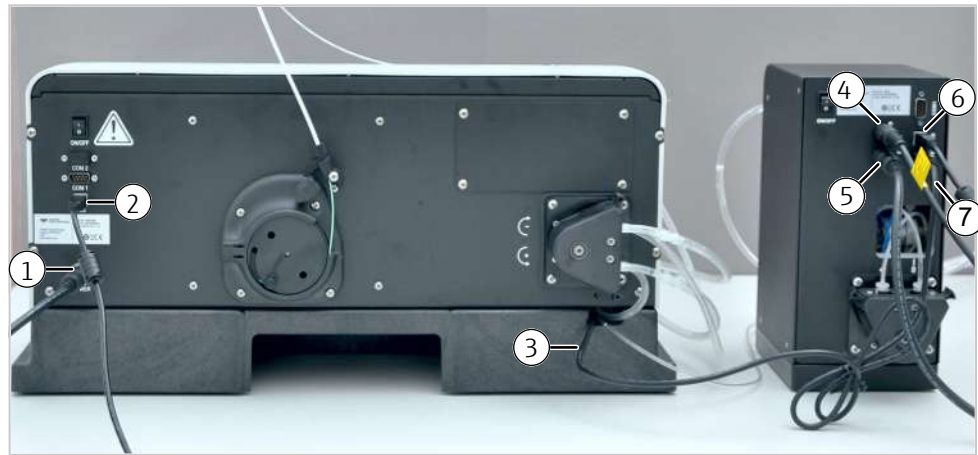


Figure 20 Connecting the sampler and the dilution system

Connections on the rear of the sampler:

- | | |
|--|---------------------------------|
| 1 Sampler power supply (via dilution system) | 2 USB interface to PC (via hub) |
| 3 Vortexer cable (to dilution system) | |

Connections on the rear of the dilution system:

- | | |
|---------------------------------|-----------------------------------|
| 4 Dilution system power supply | 5 Sampler power supply connection |
| 6 USB interface to PC (via hub) | 7 Vortexer cable connection |

- ▶ Connect the sampler and the dilution system with the control PC via a hub.

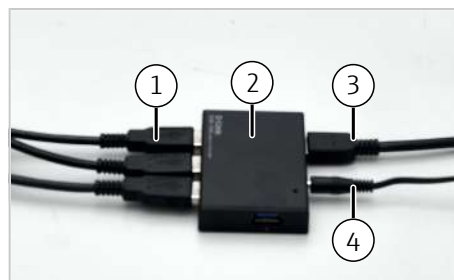


Figure 21 Control PC connection via hub

- | | |
|---|--------------------|
| 1 USB cable connections from the sampler, dilution system, etc. | 2 Hub |
| 3 USB cable to the PC | 4 Hub power supply |

- ▶ Connect the sampler and the dilution system with each other and the emission spectrometer via the following hoses:

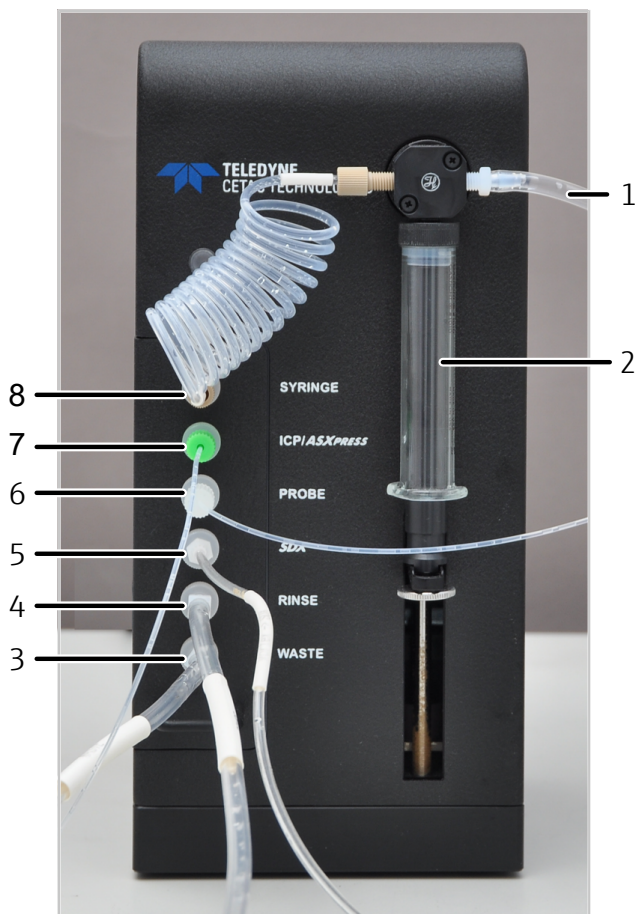


Figure 22 Hose connections on the dilution system

- | | |
|--|---|
| 1 Diluent storage bottle connection | 2 Syringe pump |
| 3 Waste bottle connection | 4 Purge liquid storage bottle connection (for vortexer mixing vessel) |
| 5 Vortexer mixing vessel connection (placed on the sampler) | 6 Sampler syringe connection |
| 7 Emission spectrometer sample hose connection (via hose pump and nebulizer) | 8 Syringe pump connection (via hose loop) |

Coupling the sampler and the dilution system with the switching valve

- ▶ Connect the sampler and the dilution system as described.
- ▶ Connect the sampler with the switching valve control unit via the RS 232 interface (COM 1).
- ▶ Connect the following on the switching valve control unit:

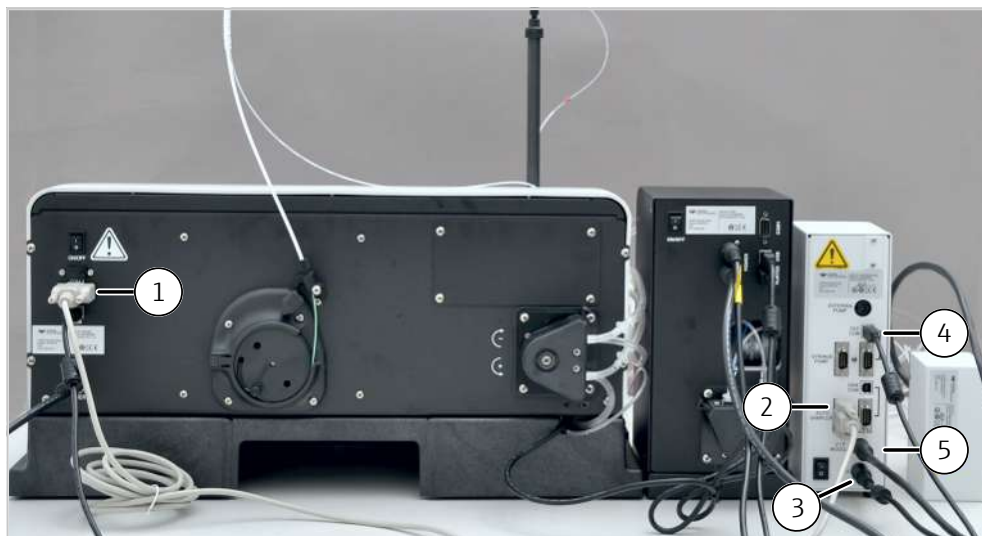


Figure 23 Connecting the switching valve control unit

Connections on the rear of the sampler:

- 1 RS 232 interface (COM 1) to the control unit

Connections on the rear of the switching valve control system:

- 2 RS 232 interface to the sampler
- 3 Control unit power supply
- 4 USB to the PC (via hub)
- 5 Interface to the switching valve

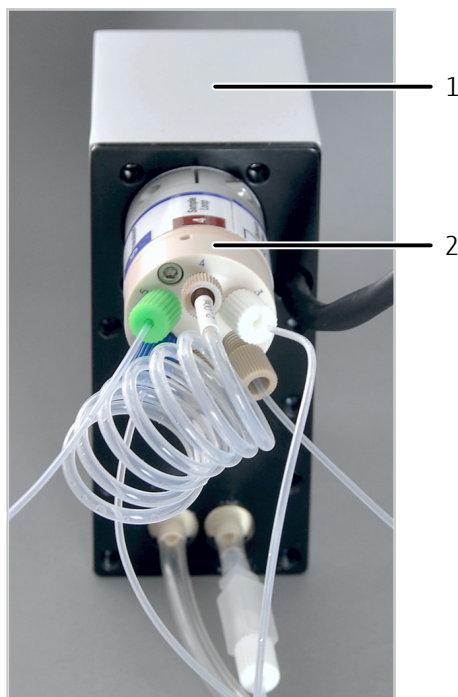


Figure 24 Connecting the hoses to the switching valve

- 1 Switching valve
- 2 6-port valve with labeled hose connections

- ▶ Connect the switching valve to the dilution system ("ICP/ASXpress" connection) via hose connection 2 ("Autosampler") on the 6-port valve.
- ▶ Connect the switching valve with the sample hose of the emission spectrometer via hose connection 5 ("Nebulizer").

Software support during installation and commissioning

Control of the sampler and the dilution system is integrated into the ASpect PQ software.

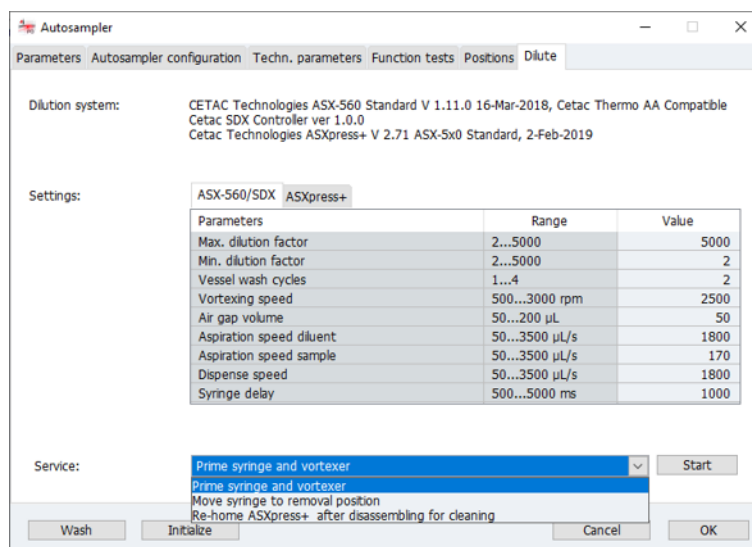


Figure 25 Autosampler page, Dilute tab

For commissioning and maintenance of the sampler and the dilution system, use the following software commands in the **Dilute** tab in the **Service** area:

Time	Measure	Software command
<ul style="list-style-type: none"> Installation of the dilution system During syringe replacement 	Inserting the syringe in the syringe pump of the dilution system. Move syringe plunger to 1/2 position for this.	Move syringe to removal position
<ul style="list-style-type: none"> Commissioning of the dilution system after installation or maintenance After changing the diluent For cleaning after use of a highly acidic or alkaline purging solution or an organic solvent 	Purging the syringe pump, hoses and vortexer mixing vessel with purging solution. Removing air bubbles from the hoses.	Prime syringe and vortexer
<ul style="list-style-type: none"> After weekly cleaning and after replacement of the 6-port valve 	Initializing the switching valve. The control electronics set the internal valve stops and determine the correct stroke.	Re-home ASXpress+ after disassembTime To Evacuate Probeling for cleaning

The operator can set the following parameters within the specified ranges in the **Autosampler** window in the **Dilute** tab:

- Max. dilution factor and Min. dilution factor
- Vessel wash cycles
- Syringe delay
- Syringe delay
- Aspiration speed diluent, Aspiration speed sample and Dispense speed
- Syringe delay

When the sampler, the dilution system and the switching valve are coupled, the operator controls the switching valve via the ASpect PQ software.

Adjust the following settings in the analysis system during switching valve commissioning: (These settings do not usually require changes during operation.)

- Loop Rinse Delay and Extra Loop Rinse
- Loop Evacuation Delay and Loop Load Time
- Equalization Delay
- Time To Evacuate Probe and Probe Wash
- Rinse Station Fill

When operating the sample and switching valve **without** the dilution system:

- ▶ Install the Dashboard software, see user manual of the accessory.
- ▶ Operate the switching valve in automatic mode (default).
- ▶ During commissioning: Adjust settings such as **Loop Rinse Delay** in the analysis system via the Dashboard software.

4.4.2 Installing the IsoMist XR temperature-controlled spray chamber



CAUTION

Risk of frostbite

The spray chamber and the inner surfaces of the Peltier element can become very cold (temperature range: -25 °C to +80 °C).

- Do not touch the spray chamber or Peltier element during or directly after operation.
-



Figure 26 Installing the temperature-controlled spray chamber

- | | |
|---------------------------------|--|
| 1 Power cable | 2 USB port |
| 3 Waste hose | 4 Sample hose on the nebulizer |
| 5 Nebulizer | 6 Temperature-controlled spray chamber |
| 7 Argon hose (to the nebulizer) | 8 Transfer tube |

- ▶ Place the temperature-controlled spray chamber in the sample chamber of the emission spectrometer.
- ▶ Connect the waste hose (3) to the port on the bottom of the Isomist.
- ▶ Connect the sample hose (4) and argon hose (7) to the nebulizer (5).
- ▶ Clamp the waste and sample pump hoses in the hose pump between two stoppers. Note the pump direction for this (see arrows).
- ▶ Insert the sample hose in the sample or connect it to the sampler.
- ▶ Insert the waste hose in the waste container.
- ▶ Connect the temperature-controlled spray chamber to the PC via the USB cable (USB port, 2). Alternately, connect the Bluetooth USB adapter to the PC.
- ▶ Attach the transfer tube (8) to the top outlet of the spray chamber.
- ▶ Connect the transfer tube to the torch via fork clamp.
- ▶ Connect the temperature-controlled spray chamber to the power grid via the power supply cable (1).
- ▶ For deinstallation: first remove the transfer tube, then move the carriage with the torch. The transfer tube can break otherwise.

4.4.3 Installing the argon humidifier

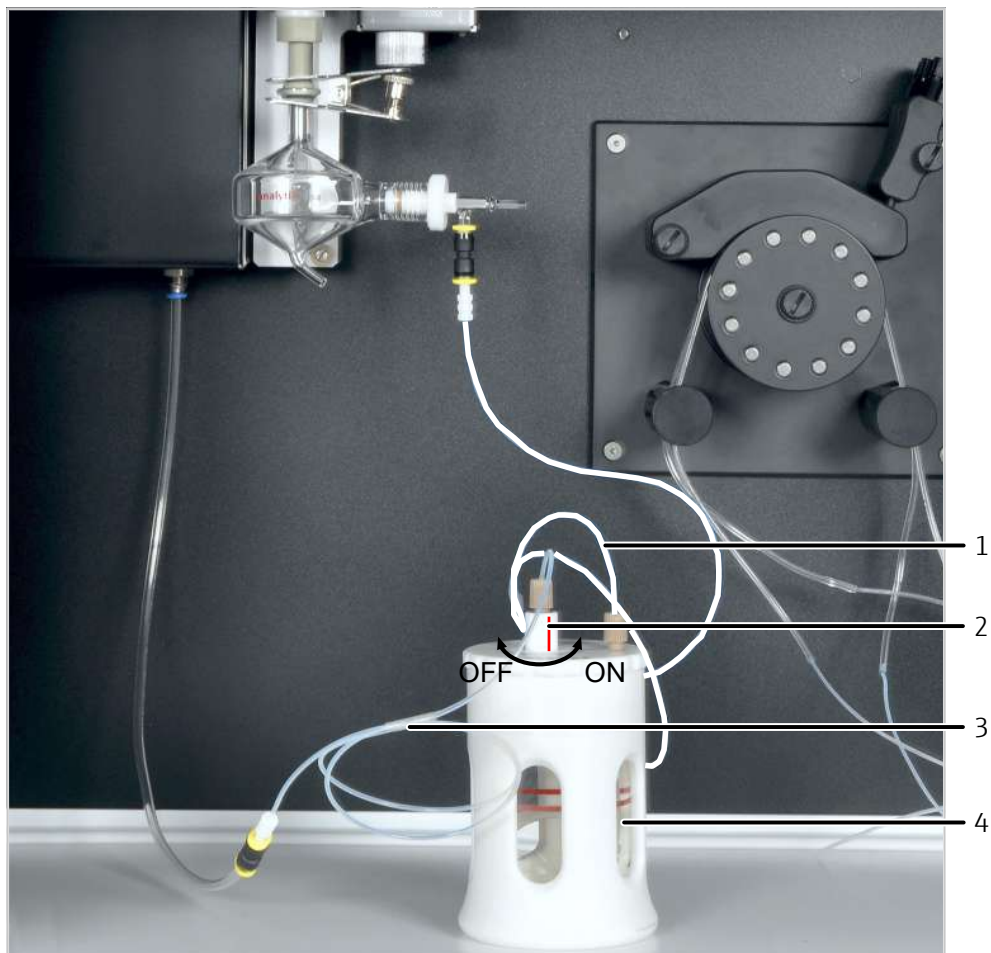


Figure 27 Installing the argon humidifier

- | | |
|---|-----------------------------------|
| 1 Gas outlet: Argon hose to the nebulizer | 2 Bypass valve |
| 3 Gas inlet: Argon hose from the ICP-OES | 4 Glass vessel with membrane coil |

- ▶ Assemble the argon humidifier as described in the accompanying datasheet. Ensure that the membrane coil is not damaged.
- ▶ Fill the glass vessel with the membrane coil (4) with deionized water up to the marking.
- ▶ Connect the hose on the gas outlet of the argon humidifier (1) with the nebulizer via a plug-in connector.
- ▶ Connect the hose on the gas outlet (3) with the argon hose of the emission spectrometer via a plug-in connector.
- ▶ Turn the bypass valve (2) until the colored marking is at "ON".

Argon humidification can be switched on and off via the bypass valve without disconnecting hose lines.

4.4.4 Installing the inline filter

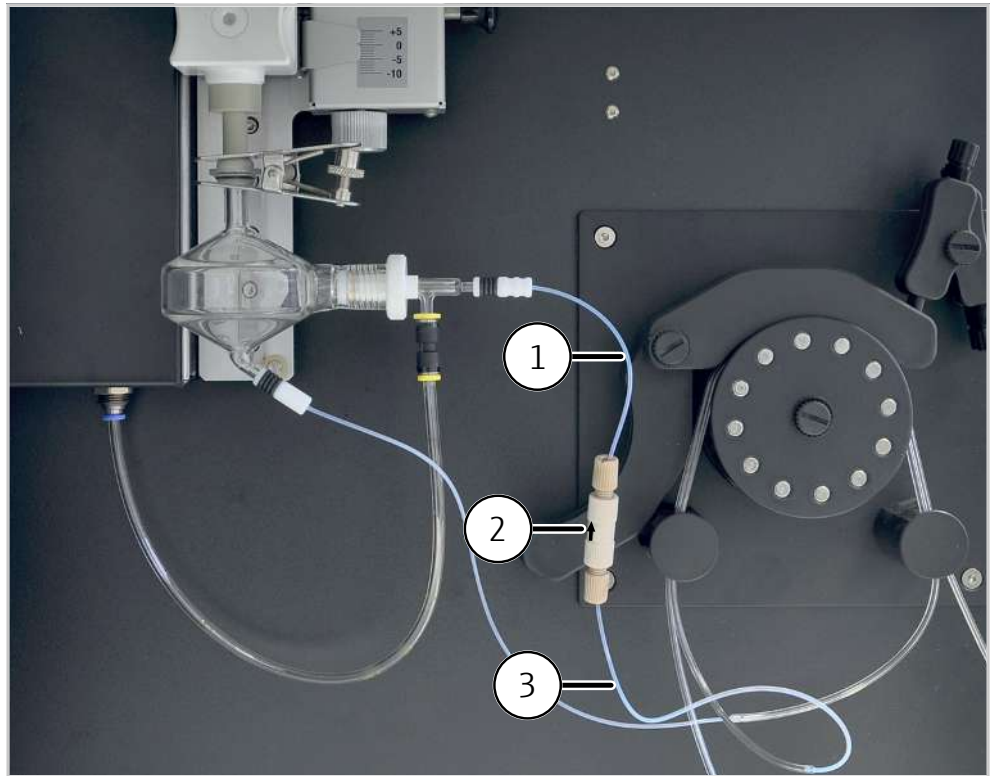


Figure 28 Installing the inline filter

- | | |
|--------------------------------|-------------------------------------|
| 1 Sample hose to the nebulizer | 2 Filter block with hose connectors |
| 3 Sample pump hose | |

- ▶ Insert the inline filter in the hoses so that the arrow on the filter block point in the flow direction (i.e. in the direction of the nebulizer).
- ▶ Assemble the inline filter as described in the accompanying datasheet.
- ▶ To do so, insert the nebulizer hose in the banjo bolt via a ferrule. The conical side of the ferrule must point toward the banjo bolt.
- ▶ Screw the hose connector into the outlet of the filter block.
- ▶ Screw a short capillary hose into the inlet of filter block via hose connector.
- ▶ Connect the capillary hose with the sample pump hose. To do this, push the capillary hose into the pump hose.
- ▶ Connect the nebulizer hose with the nebulizer.

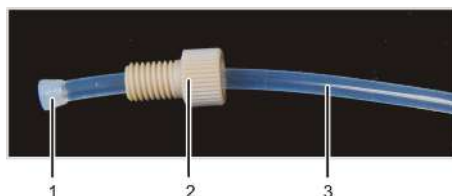


Figure 29 Fit of the ferrule in the banjo bolt

- | | |
|-----------|--------------|
| 1 Ferrule | 2 Banjo bolt |
| 3 Hose | |

5 Operation

5.1 Switching on the emission spectrometer and igniting the plasma



CAUTION

Risk of ozone and nitrous oxide poisoning



- Switch on the exhaust unit prior to igniting the plasma.
- Leave the exhaust unit switched on during operation.

Device-internal safety circuits check the following before igniting the plasma:

- The gas flow, the cooling and exhaust are switched on and comply with the specified connection requirements.
- The torch is in working position.
- The plasma compartment door is closed.

The plasma will not be ignited if a fault is present.

- ▶ Switch on the emission spectrometer via the power switch.
- ▶ Switch on the PC via the power switch and start the operating system.
- ▶ Open the gas supply and set the gases to an inlet pressure of 600 kPa (6 bar).
- ▶ Switch on the exhaust unit.
- ▶ Switch on the recirculating chiller via the power switch.
- ▶ Check that the torch is in the start position. The injector tip must be situated approx. 1 – 2 mm below the bottom edge of the induction coil.
- ▶ Check the cone of the axial observation window for dirt and wear. Check the cone for proper fit with the supplied C-wrench.
NOTICE! If the cone is loose, it will not be cooled sufficiently and will corrode.
- ▶ Close the door to the plasma compartment.
- ▶ Check the pump hoses. Replace hoses if they have lost elasticity or show strong signs of wear.
- ▶ Clamp the pump hoses into the hose pump between two stoppers.
- ▶ Place the clamping brackets over the hoses and fasten them with the clamping levers. Make sure that the clamping levers snap into place!
NOTICE! Note the pump direction! The pump rotates in a counterclockwise direction!
- ▶ Check whether there is sufficient purging solution in the bottle for the analysis. The purging solution must have the same acid content as the samples and standard solutions. Use a 2% nitric acid solution unless specified differently.
- ▶ Empty the waste bottle.
- ▶ For manual operation without a sampler: Insert the sample intake hose in the purging solution. No air must follow during the plasma ignition process.
- ▶ For manual operation: Switch on the sampler via the power switch and clamp the pump hoses to the purging pump of the sampler.
- ▶ Start the ASpect PQ program.
- ▶ Select the **Routine** or **Method development** option in the **Quick start**.



- When using the HF kit, select the **Torch material / ceramics** option to adjust the sensitivity of the optical plasma sensor.
- Optional: In the **Worksheet** area, select worksheets prepared for the quickstart, for example for analysis of elemental impurities in pharmaceutical products in accordance with USP 232/233. The worksheets contain method settings and prepared sequences.
- ▶ Exit the **Quick start** via [OK].
- ▶ If the system has been shut down for a longer period (more than a day) or the spray chamber has been dismantled, purge the spray chamber and the torch with carrier gas to evacuate any air from the sample introduction system:
 - Open the **Plasma | Control** window by clicking .
 - Click on **[Rinse spray chamber]** and wait 60 s. After this, ignite the plasma.
- ▶ Igniting the plasma:
 - Open the **Plasma | Control** window by clicking , and click on **[Ignite plasma]**. The plasma will be ignited if the coolant temperature at the coolant inlet is within the specified range (17 to 24 °C).
 - ✓ The plasma ignites.
- ▶ Check that the plasma forms correctly, i.e. the plasma extends conically beyond the induction coil and tapers toward the top.
- ▶ If a ring plasma (plasma only forms within the induction coil) forms or a rattling noise is heard: Press the red plasma deactivation switch on the left side of the device.
 - Check that the sample hose is inserted in the purging solution and that the gas supply and recirculating chiller are OK before the next ignition attempt.
 - ✓ The spectrometer is only cooled after successful plasma ignition and stable formation. The ignition process is complete after 1 to 2 min and the hose pump starts. The emission spectrometer is ready for measurement. Further settings can now be adjusted for the analysis system.

5.2 Switching off the emission spectrometer



NOTICE

Risk of damage to the torch due to high temperatures

- After the plasma has been extinguished, wait 3 min. Switch off the device via the power switch only after this time.
-
- ▶ After the end of analysis, pump purging solution through the analysis system for approx. 3 min followed by deionized water for 1 min.
 - ▶ Allow the device to run dry after this until no more liquid is in the hoses. If any hoses need to be replaced, they will be drained of acid as a consequence.
 - ▶ To extinguish the plasma by clicking on  in the toolbar.
 - ▶ Alternatively, use  to open the **Plasma** window and click on **[Plasma off]**.
 - ▶ Select the **File | Exit** menu item to close the control software.

- ▶ Confirm the prompt to switch off the purging gas for the spectrometer with **[Yes]** if it must be switched off.
- ▶ If work is only interrupted for a short period (up to 30 min.): Do not switch off the purge gas. This saves the wait time for the spectrometer to be sufficiently purged during the ignition process.
- ▶ Wait for the message that the device and the cooling can be switched off.
- ▶ Switch off the emission spectrometer, and, if applicable, the sampler via the respective power switch.
- ▶ Unclamping the pump hoses from the hose pump:
 - Release the clamping levers so that the clamping brackets no longer press on the hoses.
 - Pull the hose stoppers on the left side of the pump from the restraint.
- ▶ When using the sampler, unclamp the pump hoses in the same manner.
- ▶ Shut off the gas supply after switching off the devices.
- ▶ Switch off the recirculating chiller via the power switch.
- ▶ Switch off the exhaust unit.
- ▶ Close Windows and switch off the PC.
 - ✓ The analysis system is now switched off.

5.3 Switching off the device via the plasma deactivation switch in an emergency

Immediately switch off the plasma using the plasma deactivation switch on the left-hand side of the device if one of the following faults occur:

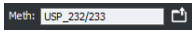
- A rattling sound can be heard
- A ring plasma forms (plasma forms only in the induction coil)
- The quartz glass of the external torch tube glows red-hot.
- No communication with the PC


Wait at least 30 s for the emission spectrometer to cool down before switching it off via the power switch.

After manually switching off the plasma or an automatic shutoff by one of the device's safety circuits: Check that all requirements for switching the device on are met before igniting the plasma again.

5.4 Starting a measurement routine

A method must be developed before a measurement can be made. The application team is on hand to offer support. Please observe the information provided in the operating instructions of the ASpect PQ program.

- ▶ Switch on the emission spectrometer and ignite the plasma.
- ▶ Select a method:
 - In the toolbar, click on the  folder symbol next to the **Method** field and select the method in the database window.
- ▶ Create or load a sequence:
 - Perform a calibration at the beginning of the sequence.

- When loading a sequence, make sure that the calibration is compatible with the method.
Analysis lines of the calibration standards must match the calibration in the method.
- After calibration, measure a QC sample in order to verify the correctness of the calibration.
- ▶ Create a sample ID table.
- ▶ Starting the measurement:
 - Start the measurement routine by clicking on  or selecting the **Routine | Start sequence** menu item.
 - In the **Start sequence** window, select or enter a file name for the results file.
 - ✓ After selection of the file name, the measurement routine will start according to the settings made in the method and the sequence.
- ▶ When using a sampler, the measurement runs automatically.
When performing sampling manually without a sampler, follow the instructions in the software on sample supply.

6 Troubleshooting

6.1 Fault messages in the software



NOTICE

Risk of device damage

- Contact the Analytik Jena GmbH customer service in the following cases:
- The troubleshooting measures described do not provide a solution for eliminating the error.
- The error occurs frequently.
- The error message is not featured in the following list or the list refers to the customer service for troubleshooting the error.

The system is monitored as soon as the device is switched on. After starting the control software, all malfunctions on the device are reported using error messages. Error messages consist of an error code and an error message.

The following section describes a number of possible malfunctions which the operator can partly troubleshoot without the help of a customer service technician. Confirm the error message and carry out the troubleshooting measures.

Fault code/fault message	
3762: Wavelength correction error!	
3765: No neon correction peak found!	
3766: Correction range exceeded!	
3782: No neon peaks found!	
3783: Too many neon peaks found!	
3783: No prim peak available!	
Cause	Remedy
<ul style="list-style-type: none"> ▪ Faulty neon or prism correction 	<ul style="list-style-type: none"> ▪ Switch the device off and on again ▪ In the event of repeated occurrence, determine which correction is faulty in the Spectrometer Parameters window in the Ne correction area. ▪ Inform customer service
3811: No factory data found in instrument storage (FINFO)!	
Cause	Remedy
<ul style="list-style-type: none"> ▪ There is no production data for line offset present in the device memory ▪ Faulty RAM memory 	<ul style="list-style-type: none"> ▪ Contact customer service for line offsets ▪ Inform customer service
3870: No purge gas available!	
Cause	Remedy
<ul style="list-style-type: none"> ▪ No Argon gas pressure 	<ul style="list-style-type: none"> ▪ Check the gas pressure ▪ Check the fit of the cone of the axial observation window
3871: No cooling water available (detector cooling)!	
Cause	Remedy
<ul style="list-style-type: none"> ▪ Recirculating chiller not switched on 	<ul style="list-style-type: none"> ▪ Switch on the recirculating chiller

Fault code/fault message	
<ul style="list-style-type: none"> Coolant flow too low 	<ul style="list-style-type: none"> Check that the coolant flow is > 0.85 l/min
3872: CCD cooling is inactive!	
Cause	Remedy
<ul style="list-style-type: none"> Plasma ignition aborted 	<ul style="list-style-type: none"> If the plasma is burning, activate the CCD cooling option in the Spectrometer window and click on [Set]
3874: Spectrometer purging is still active!	
Cause	Remedy
<ul style="list-style-type: none"> Ar purging of the spectrometer not yet complete 	<ul style="list-style-type: none"> Wait until the fault message disappears and purging is complete
4003: Plasma shut-down because emergency switch has been activated!	
Cause	Remedy
<ul style="list-style-type: none"> The red plasma deactivation switch on the left side of the device has been pressed 	<ul style="list-style-type: none"> Ignite the plasma again
4004: Plasma shut-down by plasma sensor!	
Cause	Remedy
<ul style="list-style-type: none"> Air present in the spraying chamber during ignition of the plasma Flickering and unstable plasma due to the sample matrix 	<ul style="list-style-type: none"> Before ignition, close the clamping brackets of the pump hoses, insert the hoses in water, purge the spraying chamber with Ar via nebulizer gas Dilute the sample matrix Adjust the plasma conditions
4005: Plasma shut-down! Torch positioning error	
Cause	Remedy
<ul style="list-style-type: none"> No torch installed Torch not pushed up to working position 	<ul style="list-style-type: none"> Install torch Push torch to working position
4006: Plasma shut-down because water flow is too low!	
Cause	Remedy
<ul style="list-style-type: none"> Recirculating chiller not switched on Coolant flow too low 	<ul style="list-style-type: none"> Switch on the recirculating chiller Determine cooling water flow Service the coolant
4007: Plasma shut-down! Generator error (enable)	
Cause	Remedy
<ul style="list-style-type: none"> Communication interrupted Faulty generator 	<ul style="list-style-type: none"> Restart device and PC Inform customer service
4009: Plasma shut-down because cooling water temperature is too high (in)!	
Cause	Remedy
<ul style="list-style-type: none"> Cooling temperature setting on the recirculating chiller too high 	<ul style="list-style-type: none"> Set recirculating chiller to a cooling temperature of 18 °C
4010: Plasma shut-down because cooling water temperature too high (out)!	
Cause	Remedy
<ul style="list-style-type: none"> Coolant flow too low Cooling temperature setting on the recirculating chiller too high High temperatures in the room the device is operated in have heated the water in the coolant hoses. 	<ul style="list-style-type: none"> Determine the cooling water flow, service the coolant Set recirculating chiller to a cooling temperature of 18 °C Set recirculating chiller to 18 °C. Wait until the water temperature at the device inlet is within the range of 17 to 24 °C and retry ignition.

Fault code/fault message	
4011: Plasma shut-down! Cooling water temperature!	
Cause	Remedy
<ul style="list-style-type: none"> Cooling water temperature > 25 °C (inlet) or < 22 °C (outlet) 	<ul style="list-style-type: none"> Coolant flow too low. Determine the cooling water flow, service the coolant Set recirculating chiller to a cooling temperature of 18 °C
4013: Plasma shut-down: gas flow control error (MFC)!	
4015: Argon inlet pressure too low!	
Cause	Remedy
<ul style="list-style-type: none"> No Ar gas flow present 	<ul style="list-style-type: none"> Open Ar gas cylinder Set Ar inlet pressure to 600 kPa (6 bar)
4023: Ignition failed! RF generator!	
Cause	Remedy
<ul style="list-style-type: none"> Generator shutdown due to fault during plasma generation 	<ul style="list-style-type: none"> Check sample supply Restart device
4031: Cooling water stopped because temp. is too low.	
Cause	Remedy
<ul style="list-style-type: none"> Cooling temperature settings on the recirculating chiller too low 	<ul style="list-style-type: none"> Set recirculating chiller to 18 °C. Wait until the water temperature at the device inlet is within the range of 17 to 24 °C and retry ignition.
4032: Plasma shut-down: not stable!	
Cause	Remedy
<ul style="list-style-type: none"> Unstable plasma due to sample matrix or entry of oxygen (leaks) 	<ul style="list-style-type: none"> Adjust plasma conditions (increase performance) Reduce nebulizer gas flow Reduce pump speed Increase distance of torch to cone; reduce distance to the induction coil; search for leaks in the Ar gas line, if necessary
4301: Firmware update communications error	
4302: Invalid checksum of firmware application!	
4303: Invalid firmware block!	
4304: Invalid firmware block sequence	
4305: Write-error firmware update	
Cause	Remedy
<ul style="list-style-type: none"> Firmware update failed 	<ul style="list-style-type: none"> Repeat firmware update Inform customer service
5204: Status: Plasma error!	
Cause	Remedy
<ul style="list-style-type: none"> Device communication fault Defective step motor for grating, prism, shutter 	<ul style="list-style-type: none"> Restart device (and PC if necessary) Inform customer service
5206: Status: One or more safety interlocks are open!	
Cause	Remedy
<ul style="list-style-type: none"> No cooling water flow Plasma compartment door open Torch not in measuring position 	<ul style="list-style-type: none"> Switch on the recirculating chiller. Check that the cooling water flow is > 0.85 l/min

Fault code/fault message	
<ul style="list-style-type: none"> ▪ No Ar gas pressure present ▪ Exhaust performance insufficient ▪ Manual generator shutdown via red plasma deactivation switch 	<ul style="list-style-type: none"> ▪ Close plasma compartment door ▪ Check torch position ▪ Check Ar gas pressure ▪ Check exhaust ▪ Ignite the plasma again
5208: Status: CCD cooling error! Please check purge gas flow!	
Cause	Remedy
<ul style="list-style-type: none"> ▪ No Ar gas flow 	<ul style="list-style-type: none"> ▪ If the plasma is burning, activate the CCD cooling option in the Spectrometer window and click on [Set]

6.2 Device faults and analytical problems

This section describes a number of device errors and analytic problems, some of which the user can rectify himself. Most of the device errors described are easy to identify. Most of the analytic problems lead to implausible measurement results. If the suggested solutions do not eliminate the errors/problems, and if such problems occur frequently, contact the customer service department of Analytik Jena GmbH.

No signal	
Cause	Remedy
<ul style="list-style-type: none"> ▪ Pump supplies no sample 	<ul style="list-style-type: none"> ▪ Check hose/hose pump
<ul style="list-style-type: none"> ▪ Nebulizer clogged, mass-flow rate very high (in the Plasma report window) 	Check with Na solution (1 g/L), if necessary. If no plasma coloration (orange) is observed: <ul style="list-style-type: none"> ▪ Check that nebulizer nozzles are unclogged and clean nebulizer ▪ If sample nozzle is clogged, filter solutions or use inline filter ▪ If argon nozzle is clogged, dilute measuring solutions or use argon humidifier
<ul style="list-style-type: none"> ▪ Injector clogged 	Check with Na solution (1 g/L). If no plasma coloration (orange) is observed: <ul style="list-style-type: none"> ▪ Check injector tip for deposits and clean it ▪ Increase distance between injector and plasma (move torch down on height adjustment or increase auxiliary gas flow) ▪ Use argon humidifier or inline filter
<ul style="list-style-type: none"> ▪ Nebulizer gas set too low 	<ul style="list-style-type: none"> ▪ Optimize carrier gas flow
<ul style="list-style-type: none"> ▪ Adjustment on analyte channel 	<ul style="list-style-type: none"> ▪ Adjust method parameter x/y offset in the Spectrometer Adjust plasma view window (see online help or software instructions)
<ul style="list-style-type: none"> ▪ Leak in the sample supply system (sample and pump hoses) 	<ul style="list-style-type: none"> ▪ Check sample and pump hoses and their plug connectors
<ul style="list-style-type: none"> ▪ Plasma compartment windows dirty 	<ul style="list-style-type: none"> ▪ Replace windows
<ul style="list-style-type: none"> ▪ Transparency in the UV vacuum missing 	<ul style="list-style-type: none"> ▪ Check the duration of the purging gas flow. Wait for the purging gas to completely flood the spectrometer chamber
Sensitivity too low	
Cause	Remedy
<ul style="list-style-type: none"> ▪ Same fault causes and remedies as the "No signal" fault screen 	

Measured value too low	
Cause	Remedy
<ul style="list-style-type: none"> Incorrect calibration 	<ul style="list-style-type: none"> Check calibration solutions and repeat calibration
<ul style="list-style-type: none"> Low solubility substances lead to low concentrations Low solubility substances not completely digested 	<ul style="list-style-type: none"> Optimize sample preparation
<ul style="list-style-type: none"> Volatile substances escape during sample preparation 	<ul style="list-style-type: none"> Optimize sample preparation
<ul style="list-style-type: none"> Spectral interference in the calibration standard 	<ul style="list-style-type: none"> Use another analytical line
<ul style="list-style-type: none"> Background correction fault 	<ul style="list-style-type: none"> Select background correction points that are not spectrally disturbed Better adjustment of a curved background via non-linear correction function
<ul style="list-style-type: none"> Fault when using an internal standard 	<ul style="list-style-type: none"> Wrong standard dosage Internal standard concentration not in linear range. Select lower concentration for standard Insufficient reaction adjustment to plasma temperature change. Matrix adjustment and better fit between the analytical line behavior and the internal standard
<ul style="list-style-type: none"> Contamination/carry-over in the calibration solution 	<ul style="list-style-type: none"> Remedy carry-over/contamination cause
<ul style="list-style-type: none"> Viscous sample solution/higher density than calibration solution 	<ul style="list-style-type: none"> Matrix adjustment (add to calibration solutions or dilute) Use one/several internal standards
<ul style="list-style-type: none"> Spray chamber filled 	<ul style="list-style-type: none"> Empty spray chamber Check drainage pump hose, select larger diameter if necessary
Measured value too high	
Cause	Remedy
<ul style="list-style-type: none"> Calibration fault 	<ul style="list-style-type: none"> Check calibration solution
<ul style="list-style-type: none"> Peak position slightly shifted or non-peak measurement 	<ul style="list-style-type: none"> Spectral interference overlooked. Use another analytical line or activate fault correction
<ul style="list-style-type: none"> Contamination/carry-over 	<ul style="list-style-type: none"> Find and remedy cause for contamination/carry-over
<ul style="list-style-type: none"> Concentrations appear higher due to volatile substances 	<ul style="list-style-type: none"> Optimize sample preparation
<ul style="list-style-type: none"> Analyte is an alkaline metal (or an easily excitable atomic line) 	<ul style="list-style-type: none"> Alkaline effect. Optimize plasma temperature (nebulizer gas flow and/or performance) and plasma observation
<ul style="list-style-type: none"> Fault when using an internal standard 	<ul style="list-style-type: none"> Wrong standard dosage Insufficient reaction adjustment to plasma temperature change. Matrix adjustment and better fit between the analytical line behavior and the internal standard
<ul style="list-style-type: none"> Warm-up phase not observed 	<ul style="list-style-type: none"> Wait for warm-up phase before calibration

<ul style="list-style-type: none"> Sample foams when shaken 	<p>Surface active substances in the measurement solutions:</p> <ul style="list-style-type: none"> Optimize sample preparation Add surface active substances to calibration solutions as well
Poor precision	
Cause	Remedy
<ul style="list-style-type: none"> Pump fast mode was active shortly before the measurement 	<ul style="list-style-type: none"> Limit fast mode to the time necessary to transfer measurement solution to nebulizer
<ul style="list-style-type: none"> Pre-purge time too short 	<ul style="list-style-type: none"> Extend pre-purge time
<ul style="list-style-type: none"> Nebulizer or injector clogged 	<p>Check with Na solution (1 g/L), if necessary. If no plasma coloration (orange) is observed:</p> <ul style="list-style-type: none"> Check that nebulizer nozzles are unclogged and clean nebulizer If sample nozzle is clogged, filter solutions or use inline filter If argon nozzle is clogged, dilute measuring solutions or use argon humidifier
<ul style="list-style-type: none"> Nebulizer gas flow not optimal 	<ul style="list-style-type: none"> Optimize nebulizer gas flow
<ul style="list-style-type: none"> Argon supply leaks 	<ul style="list-style-type: none"> Seal leaks
Signal drift	
Cause	Remedy
<ul style="list-style-type: none"> Temperature change in the spray chamber 	<ul style="list-style-type: none"> A change of 1 °C causes a drift of approx. 1 % Use temperature-controlled spray chamber or ensure air conditioning in the laboratory
<ul style="list-style-type: none"> Insufficient transparency in the UV vacuum 	<ul style="list-style-type: none"> Check if the argon purging of the spectrometer is complete (activate the purge gas flow long enough)

7 Maintenance and care

The operator may not undertake any service or maintenance work to this device and its components other than that specified in these instructions.

Observe the information in the "Safety instructions" section for all maintenance work. Compliance with the safety instructions is a prerequisite for the error-free operation of the device. Always observe all warnings and instructions that are displayed on the device itself or indicated by the control software.

To ensure faultless and safe functioning, Analytik Jena recommends an annual inspection and servicing by its Service department.



WARNING

Risk of electric shock

- Always switch off the device and disconnect the power plug before performing maintenance work.
Power to the device is only disconnected by disconnecting the power plug. After the device is switched off, some parts are still live.
- Only leave the device and the software on as the maintenance instructions explicitly demand.



CAUTION

Risk of damage to skin and eyes due to UV and electromagnetic radiation

Plasma emits UV radiation and high-frequency electromagnetic radiation which can cause serious eye and skin injuries as well as other health problems.

- Do not bypass the safety circuits with any maintenance work.
- Check the function of the safety circuits after completing maintenance work.



CAUTION

Risk of burns from the hot torch

Plasma is extremely hot. The torch is still very hot after the plasma has been extinguished. Contacting these hot surfaces can cause burns.

- Wait 5 minutes after extinguishing the plasma. Only touch the torch after this time.
-

7.1 Maintenance overview

Basic device

Maintenance interval	Maintenance task
Daily and after maintenance	<ul style="list-style-type: none"> ▪ Check the filling level of the purging solution bottle, fill as required ▪ Check waste bottle filling level, empty as required ▪ Remove contamination from sample chamber and plasma compartment ▪ Check transfer optics windows in plasma compartments for corrosion and contamination. Clean or replace as necessary. ▪ Check pump hose tightness and elasticity
Monthly	<ul style="list-style-type: none"> ▪ Check air filter on device rear for soiling, replace as necessary ▪ Check water filter in cooling water circuit for contamination, replace as necessary, at least once per year
As required	<p>Replace windows for beam entry and exit in the plasma compartment:</p> <ul style="list-style-type: none"> ▪ If streaks and burnt-in residue is visible ▪ When energy losses occur <p>Check gas connections for leaks:</p> <ul style="list-style-type: none"> ▪ After renewing connections ▪ If the manometer indicates a significant pressure loss ▪ If the plasma fails to ignite or is accompanied by loud noise <p>Replace argon hose if hose is discolored.</p>

Sample supply system

Maintenance interval	Maintenance task
As required	<ul style="list-style-type: none"> ▪ Clean torch if visibly contaminated (in particular, a metallic film or heavy milky-white discoloration of the quartz glass). Intervals can be from daily to yearly, depending on the sample material. ▪ Clean nebulizer if repeatability declines significantly for no other reason or if the baseline drifts. Contamination occurs most often with samples with high salt contents or with suspended particles. ▪ Replace the glass body of the dismantlable torch if cracked.

Sampler

Maintenance interval	Maintenance task
Daily and after maintenance	<ul style="list-style-type: none"> ▪ Clean surfaces ▪ Remove residual liquid from the tray ▪ Check sample hose and cannula for deposits ▪ Check pump hoses for tightness and elasticity, replace as necessary
Weekly	<ul style="list-style-type: none"> ▪ Clean purging vessel

Recirculating chiller

Maintenance interval	Maintenance task
Weekly and after maintenance	<ul style="list-style-type: none"> ▪ Check coolant level at the filling level indicator, refill as necessary
Every six months	<ul style="list-style-type: none"> ▪ Check cooling water conductivity
Yearly	<ul style="list-style-type: none"> ▪ Replace coolant yearly and if conductivity rises above 50 to 200 $\mu\text{S}/\text{cm}$

7.2 Basic device maintenance

7.2.1 Cleaning the dismantleable torch



WARNING

Risk of chemical burns from aqua regia

Aqua regia is a 3:1 mixture of concentrated hydrochloric acid and nitric acid. Aqua regia is highly corrosive and has an oxidizing effect.

- Wear protective glasses and clothing when creating and working with aqua regia. Work under an extraction hood.
 - Observe all instructions and specifications in the safety data sheets for the basic materials.
-



CAUTION

Risk of burns from the hot torch

Plasma is extremely hot. The torch is still very hot after the plasma has been extinguished. Contacting these hot surfaces can cause burns.

- Wait 5 minutes after extinguishing the plasma. Only touch the torch after this time.
-



CAUTION

Risk of injury

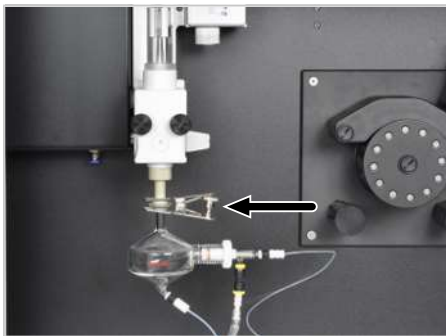
A risk of injury due to broken glass is present when handling glass parts.

- Handle glass parts with extreme caution.
-

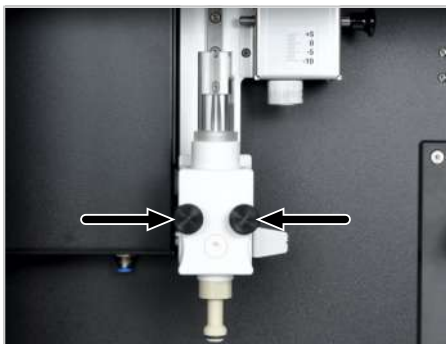
Clean the torch in case of visible contamination (deposits or scaling). Depending on the sample matrix, this can be necessary daily or at much greater intervals (monthly).



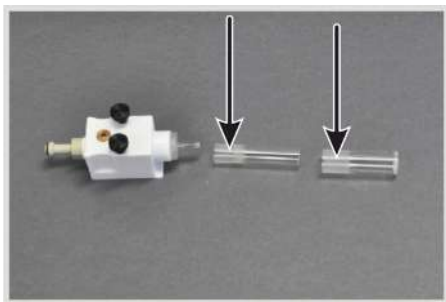
- ▶ Pull out the spring bolt on the height adjustment and carefully allow the carriage with the torch to slide down the adjusting rail.



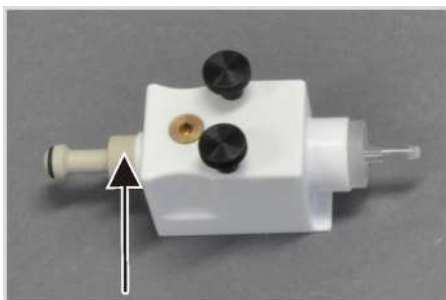
- ▶ Remove the fork clamp and remove the spray chamber.
- ▶ Carefully place the spray chamber to one side.



- ▶ Unscrew the torch from the carriage on the rail guide.



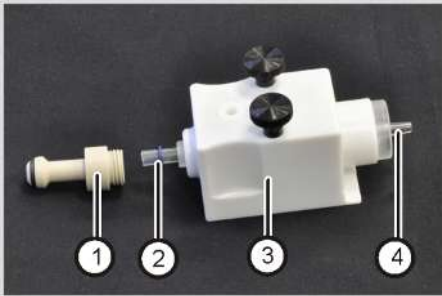
- ▶ First release the outer tube, and then the inner tube, from the holder with a rotating motion.
CAUTION! The quartz tubes are very fragile and are firmly mounted in the ground glass connector of the holder. Wear protective gloves for working with glass when disassembling the torch.



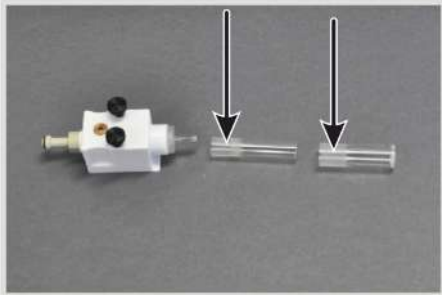
- ▶ Unscrew the connection piece from the holder. Release the injector with a rotating motion.



- ▶ Remove the quartz bonnet from the induction coil.
- ▶ Place all glass components in aqua regia for approx. 12 h.
- ▶ Rinse the glass components under deionized water (<math><1 \mu\text{S}/\text{cm}</math>) and dry them with compressed air or argon.



- ▶ Push the O-ring (2) about 1 cm onto the wide side of the injector.
- ▶ Push the injector (4) into the holder (3) with a rotating motion. Screw the connection piece (1) all the way in. This will seal and adjust the injector properly.



- ▶ Grease the joint of the inner and outer tubes with the supplied oiled cloth until it shines transparently.



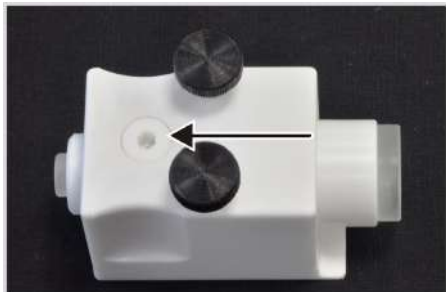
- ▶ Carefully push the inner tube into the glass body in the holder as far as it will go. Lightly rotate the tube while doing so to avoid jamming and to make sure that the ground glass joint is sealed.
- ▶ The injector tip must be exactly flush with the outer edge of the inner tube. The injector tip must not protrude beyond the outer edge of the inner tube. The tip can, however, be a maximum of 1 mm below the outer edge.
- ▶ If the injector tip could not be properly aligned:
 - Remove the inner tube from the holder and loosen the screw connection of the connection piece.
 - Push the injector into the holder as far as it will go. Slight resistance from the O-ring seal must be overcome.
 - Afterwards reinsert the inner tube and check the proper fit of the injector.



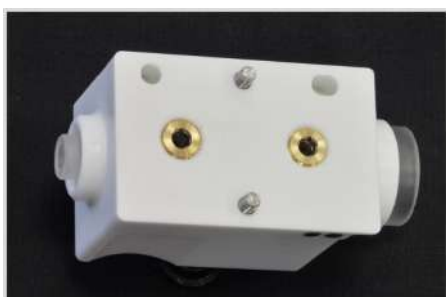
- ▶ Insert the outer tube in the glass body with a rotating motion. Ensure that the ground joint seal is tight.
- ▶ Reattach the torch and the bonnet.

7.2.2 Replacing the glass

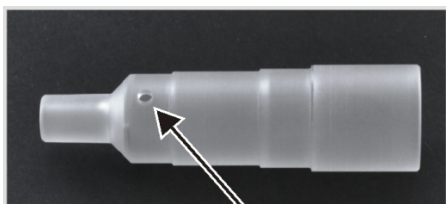
The glass body of the dismantlable torch only needs to be replaced if it is cracked. Check the glass for particle or solvent contamination when cleaning the torch. Clean the glass body as necessary.



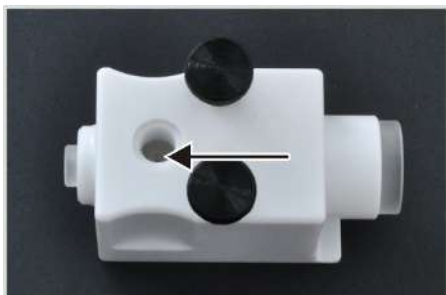
- ▶ Disassemble the torch as described.
- ▶ Unscrew the white Allen screw on the front of the holder which initially secures the glass body in the correct position.



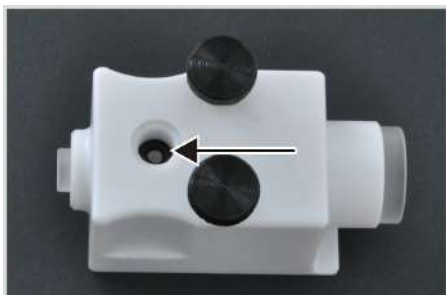
- ▶ Unscrew both connections for the argon supply on the rear of the holder.
- ▶ Pull the glass body from the holder. Remove any shards if necessary.
- ▶ Press the O-rings out of the holder.
- ▶ Clean the holder of dust and deposits.



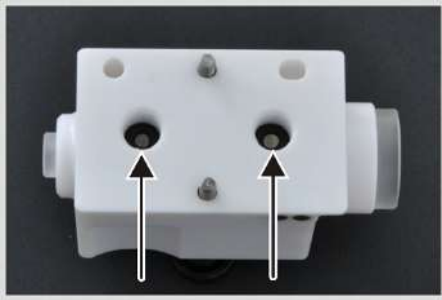
- ▶ Insert a new glass body into the holder. Align the glass body to ensure that the individual bore is visible centered in the front opening of the holder.



- ▶ The glass body is aligned correctly if the two slanted bores for the argon inlet are located at the center of the openings on the rear of the holder.



- ▶ Check the O-rings and replace any worn rings.



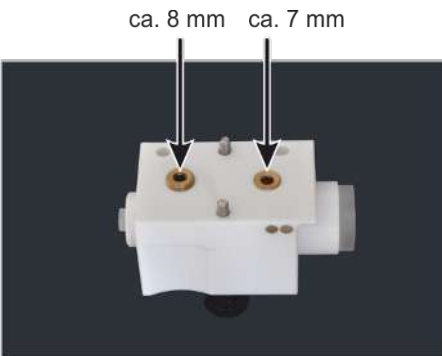
- ▶ Place O-rings in all three screw holes and carefully press onto the glass body. One O-ring is located on the front of the holder, two O-rings on the rear.



- ▶ Screw the white Allen screw into the front opening until it protrudes approx. 1 mm beyond the surface of the holder. The O-ring must not yet press against the glass body. The closure pin must protrude into the bore of the torch body, pre-centering the torch body.



- ▶ Align the top slanted hole for the argon inlet centered with the top O-ring.



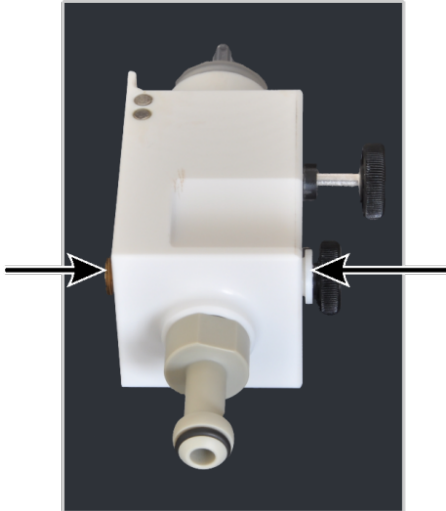
- ▶ Screw the shorter gas connection (approx. 7 mm) into the top opening to be flush with the surface of the holder.
- ▶ Screw the longer gas connection (approx 8 mm) into the bottom opening to protrude 1 mm beyond the surface of the holder.
NOTICE! The gas connections are of different lengths and must not be confused. Only screw in the gas connections until they are flush with the surface of the holder. Otherwise the glass body can break when the gas connections are screwed in!.
- ▶ Visually check the alignment of the two slanted holes through the screwed in gas connections again.




- ▶ Insert the O-ring into the connection piece and press it into the recess.
- ▶ Press the injector all the way into the connection piece with a rotating motion. The clearly noticeable resistance of the O-ring must be overcome.



- ▶ Push the injector into the glass body with a rotating motion. Push the connection piece into the holder as far as it will go. When screwing this in, only the friction resistance of the thread must be noticeable. No pressure must be applied to the glass body.



- ▶ Alternately screw in the gas connection (8 mm) and the white Allen screw by half-turns. The gas connection must end flush with the upper edge of the holder. The Allen screw must protrude slightly.
- ▶ To check, slightly unscrew the connection piece and screw it back in.
- ▶ If there is noticeable resistance against the glass body, unscrew the gas connection and Allen screw by approx. 1 mm and repeat screwing them in alternately.
- ▶ Mount the inner and outer tube (→ "Cleaning the dismantlable torch"  58).

7.2.3 Maintenance of the one-piece torch



WARNING

Risk of chemical burns from aqua regia

Aqua regia is a 3:1 mixture of concentrated hydrochloric acid and nitric acid. Aqua regia is highly corrosive and has an oxidizing effect.

- Wear protective glasses and clothing when creating and working with aqua regia. Work under an extraction hood.
- Observe all instructions and specifications in the safety data sheets for the basic materials.



CAUTION

Risk of burns from the hot torch

Plasma is extremely hot. The torch is still very hot after the plasma has been extinguished. Contacting these hot surfaces can cause burns.

- Wait 5 minutes after extinguishing the plasma. Only touch the torch after this time.



CAUTION

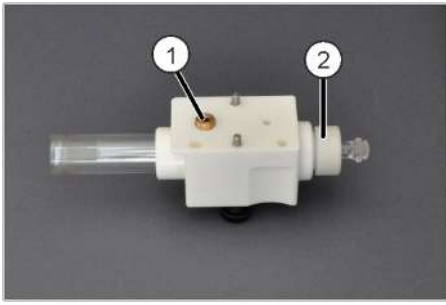
Risk of injury

A risk of injury due to broken glass is present when handling glass parts.

- Handle glass parts with extreme caution.

Cleaning the one-piece torch

The torch must be cleaned in case of visible contamination.



- ▶ Pull out the spring bolt on the height adjustment and carefully allow the carriage with the torch to slide down the adjusting rail.
- ▶ Remove the fork clamp and remove the spray chamber. Carefully place the spray chamber to one side.
- ▶ Unscrew the torch from the carriage (→ "Cleaning the dismantlable torch" 58).
- ▶ Slightly unscrew the stopper from the gas connection (1).
- ▶ Loosen the clamp screw on the torch holder (2) by one turn.



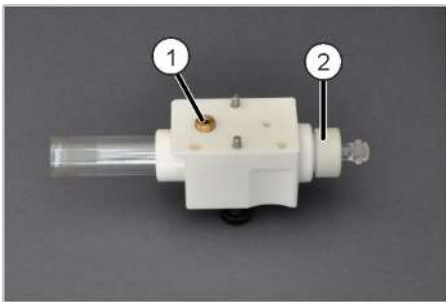
- ▶ Carefully pull the one-piece torch out of the holder with a rotating motion.
NOTICE! The torch may be firmly stuck in the holder. Wear glass handling gloves to grip the torch safely. When pulling out the torch, ensure it does not become jammed.



- ▶ Remove the quartz bonnet from the induction coil.
- ▶ Place the glass components in aqua regia for approx. 12 h.
- ▶ Rinse the glass components under deionized water (<math><1 \mu\text{S}/\text{cm}</math>) and dry them with compressed air or argon.



- ▶ Push the torch into the holder as far as it will go. At the same time, turn it so that the gas inlet opening of the torch is centered in the opening for the gas connection (arrow) of the holder.



- ▶ Screw the stopper into the gas connection (1).
NOTICE! The upper edge of the stopper must be flush with the upper edge of the holder. Do not screw it in any further under any circumstances.
- ▶ Firmly screw the clamp screw (2) into the holder. To ensure gas tightness in the lower part of the torch, the gap between the clamp screw and the holder must not exceed 0.5 mm in width.

Replacing the sealing rings

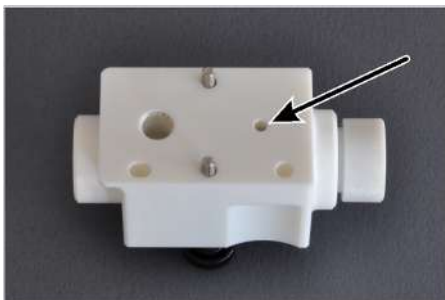
If the torch is not gas-tight, i.e., problems occur during plasma ignition, the sealing rings must be checked and replaced if necessary.



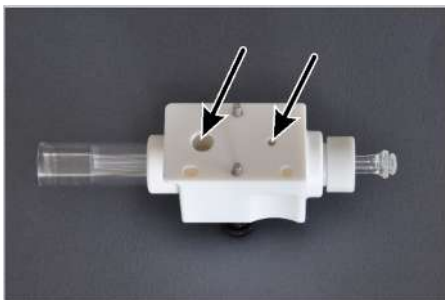
- ▶ Remove the torch from the torch holder as described.
- ▶ Unscrew the stopper from the gas connection and remove the O-ring.



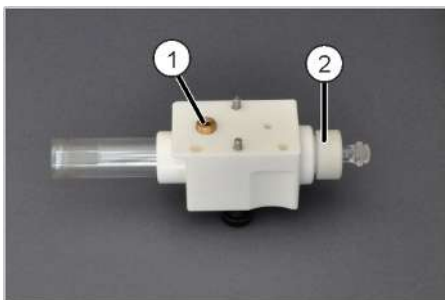
- ▶ Unscrew the clamping ring from the torch holder and remove the retaining ring, 2 O-rings and the spacer ring for the gas line.
- ▶ Check the sealing rings (O-rings) and replace any worn rings.



- ▶ Reinsert the sealing rings and the distance ring into the lower opening of the torch holder in the following order:
Green O-ring – spacer ring – green O-ring – flat retaining ring – clamping screw
 - The spacer ring must be turned so that one of the two bore holes in the ring is aligned with the small gas inlet opening (see arrow) in the torch holder.



- ▶ Push the torch into the holder as far as it will go. At the same time, turn it so that the gas inlet openings of the torch are centered in the openings for the gas connection of the holder (arrows).




- ▶ Insert the small O-ring into the gas connection.
- ▶ Screw the stopper into the gas connection (1).
NOTICE! The upper edge of the stopper must be flush with the upper edge of the holder. Do not screw it in any further under any circumstances.
- ▶ Firmly screw the clamp screw (2) into the holder. To ensure gas tightness in the lower part of the torch, the gap between the clamp screw and the holder must not exceed 0.5 mm in width.

7.2.4 Cleaning the nebulizer

Clean the nebulizer if it is clogged due to particles or high concentrations of salt in the samples. An indicator that the nebulizer has clogged up is increased carrier gas pressure.

Checking the carrier gas pressure

- ▶ Open the **Plasma | Control** window via .
- ▶ Compare the current carrier gas (pressure) percentage with the value previously achieved after installation of the new or cleaned nebulizer.
- ▶ Clean the nebulizer as described below if the percentage has increased significantly (i.e. by more than half the original value), but at a value of 75% at the latest.

Cleaning the nebulizer

Clean the nebulizer using the nebulizer cleaning tool. This tool can be ordered from Analytik Jena GmbH.

A special nebulizer cleaning tool is available for the PFA nebulizer (HF Kit) and the optional parallel-path nebulizer.

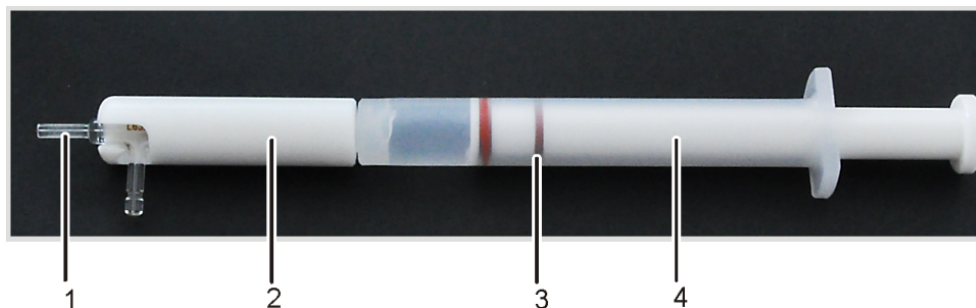


Figure 30 Nebulizer cleaning tool

- | | |
|------------------|--------------------|
| 1 Nebulizer | 2 Nebulizer holder |
| 3 1st red O-ring | 4 Syringe |



WARNING

Risk of methanol poisoning

Methanol is poisonous if inhaled, swallowed or on skin contact. The liquid and its vapors are highly flammable.

- Wear protective glass and clothing when working with methanol. Work under an extraction hood.
 - Keep methanol away from heat, sparks, open flames and hot surfaces.
 - Observe all notes and specifications in the safety data sheet.
-
- ▶ Unscrew the nebulizer holder from the syringe and fill the syringe with methanol. Pull the plunger out to the first red O-ring.
 - ▶ Screw the nebulizer holder onto the syringe.
 - ▶ Push the nebulizer with the tip first into the holder until the side carrier gas connection comes to rest in the holder groove.
 - ▶ Hold the nebulizer cleaning tool over a receptacle and push the plunger into the syringe. The methanol should flow out of both connection pieces.
 - ▶ To remove particle deposits from the nebulizer cannula: Increase the pressure by closing the carrier gas connector with a finger. Use the same method to increase the pressure by closing the sample inlet to remove particles from the carrier gas connector.

- ▶ Gently shake the nebulizer cleaning tool to remove the methanol from the nebulizer.
- ▶ Remove the nebulizer from the holder. Shake any remaining methanol from the nebulizer cleaning tool.
- ▶ Place the nebulizer in the holder once again and quickly move the plunger in and out three times to also remove the methanol from the nebulizer.
- ▶ Remove the nebulizer from the holder. Connect the nebulizer to the spray chamber. Let argon flow through the nebulizer for at least three minutes before using it for the next analysis.

7.2.5 Cleaning the sample chamber and the plasma compartment

Clean the sample chamber and the plasma compartment daily with a damp (not dripping!) cloth. A commercial surfactant can be used to remove stubborn contamination.

Remove spots, drops or larger reagent spillages and clean them using an absorbent material such as cotton wool, laboratory wipes or cellulose.

7.2.6 Checking the gas system for leaks

Check for leaks weekly or before putting the system into operation after disconnection of the device from the gas supply system. To do this, close the shut-off valve of the gas supply system and check the pressure on the downstream manometer. If the pressure drops sharply, search for leaks in the gas supply.

- ▶ Open the shut-off valve.
- ▶ Brush connections with a heavily foaming liquid (e.g., soap solution). If foam bubbles form at the gas connections when starting up, cut off the gas supply.
- ▶ Check the gas connections for proper fit. Unscrew the oxygen connection and check the sealing ring. Replace worn-out sealing rings.
- ▶ Reinsert the hose in the gas connection, ensuring proper fit, and check for leaks again.

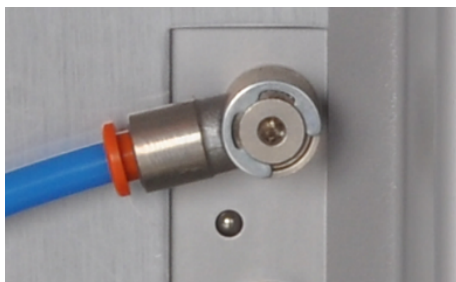


Figure 31 Plug-in connectors for gas connections

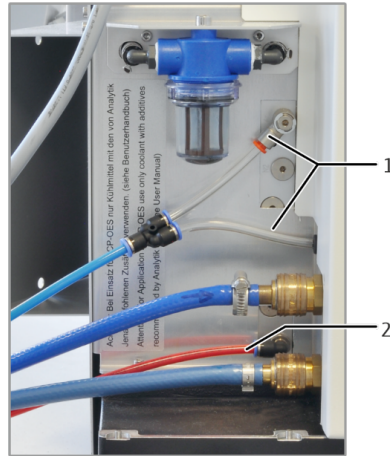


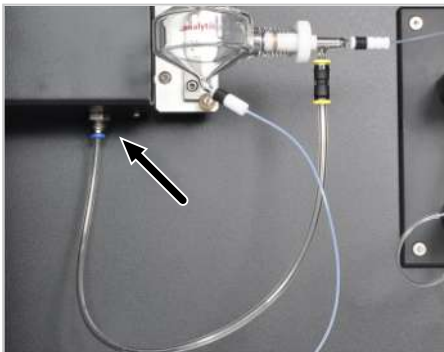
Figure 32 Connections for argon and oxygen

1 Argon

2 Oxygen

7.2.7 Replacing the argon hose

The argon supply hose for the nebulizer may become discolored. In this case, the hose must be replaced.



- ▶ Press the colored ring on the plug connector up and pull the hose down.
- ▶ Insert the new hose into the connection.

7.2.8 Changing the plasma compartment windows

The windows in the plasma compartment in front of the transfer optics must be replaced if their transmission has greatly deteriorated, especially in the UV range. Cleaning the windows does not usually completely restore UV transmission. The cleaning effect varies depending on the wavelength. Average losses of around 30% must be expected in the UV vacuum. In the visible spectrum, transparency can generally be fully restored.



NOTICE

Risk of damaging the quartz windows due to sweat from hands and ultrasound

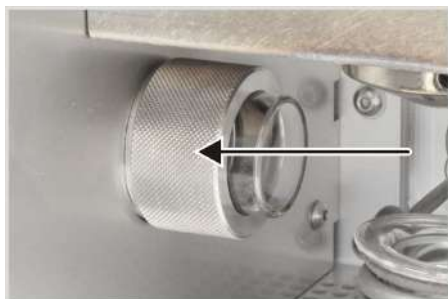
Fingerprints can burn into the surface of the quartz windows, reducing visibility.

- Do not touch the fronts of the quartz windows with your fingers. Wipe off any fingerprints immediately with ethanol.
- Do not clean the quartz windows in an ultrasonic bath. This may lower the UV permeability of the windows.

- Cleaning the windows
- ▶ Clean the windows with a cotton pad using water and a commercial surfactant. Optionally, clean the windows using aqua regia. Observe all safety information on handling this concentrated acid.
 - ▶ Rinse with water.
 - ▶ Dry via gas flow (argon or compressed air).

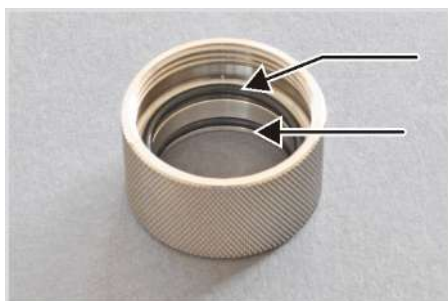
- Checking permeability
- ▶ Select a routine method.
 - ▶ Select 3 lines, one line each in the low UV range, and in the medium, and in the high wavelength range.
 - ▶ For a QC sample, determine the intensities at these 3 wavelengths and record the results in a QC card or a table.
 - ▶ If the required detection limits are no longer achieved, clean or replace the windows.

Cleaning and replacing the horizontal window The horizontal window is used for radial observation.



- ▶ Before cleaning: Activate the quick purge of the optics in the via the ASPECT PQ software in the **Spectrometer** window in the **Parameters** tab via the **[On]** button. Purging prevents contamination of the spectrometer with laboratory air. If possible, switch off the laboratory exhaust system during cleaning.

- ▶ Unscrew the window holder in a counterclockwise direction.
- ▶ Press the window out of the holder.
- ▶ Clean the window as necessary:
 - Use a cotton pad with water and a commercial surfactant.
 - Rinse with water and dry in a gas flow (argon or compressed air).



- ▶ Check the sealing rings for wear and replace as necessary.



- ▶ Insert the new or cleaned window into the holder. See the note on the fit of the window below. Do not touch the front sides with your fingers.
- ▶ Screw the holder into the plasma compartment opening.

Note on the fit of the horizontal window:

- The window can be inserted variably in its holder.
- Slide the window to the rear as far as possible to prevent the window from fogging up due to plasma as far as possible.

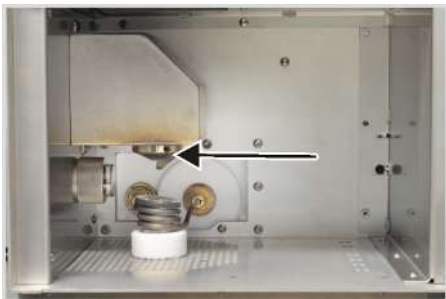
- Only slide the window as close to the torch as possible if you want to achieve the lowest possible detection limits in the vacuum UV with radial observation. This, however, presents the risk of the window fogging up more quickly, causing a drift as a result.

Replacing the window in the cone

The window in the cone is used for axial observation.



- ▶ Before cleaning: Activate the quick purge of the optics in the via the AS-pect PQ software in the **Spectrometer** window in the **Parameters** tab via the **[On]** button. Purging prevents contamination of the spectrometer with laboratory air. If possible, switch off the laboratory exhaust system during cleaning.
- ▶ Move the torch downwards out of the working position.
- ▶ Remove the quartz bonnet from the induction coil. These precautions prevent the glass parts from becoming damaged during installation of the cone.



- ▶ Clean the cone with a damp cloth and dry it.
- ▶ Unscrew the cone with the supplied hook wrench. If the window is stuck in its frame, see the description below.
- ▶ Close the opening to the optics during cleaning, for example with another cone, to prevent contamination of the optics.



- ▶ Clean the window as necessary.
- ▶ Insert a new or cleaned window into the cone and fit a sealing ring.
- ▶ Replace the worn sealing ring.
- ▶ Screw the cone firmly back into the cone opening in the plasma compartment. **NOTICE!** If the cone is loose, it will not be cooled sufficiently and will corrode quickly.

If the window is stuck in its frame:

- ▶ Hold a gloved hand underneath the cone opening.
- ▶ Carefully insert a fingernail of the second hand (with glove) or a stick (wood or plastic) into the gap between the window and the frame and lever the window out. The window falls out downward.
- ▶ Catch the windows as it falls out.
- ▶ Remove the sealing ring from the frame.

7.2.9 Replacing fuses

If a fuse is damaged, a red lamp will light up on the fuse holder.

Only use fuses of the following type:

Fuse	Type	Protected circuit
S1	10 A NFC 10x38 gG AC, 400 V	Spectrometer
S2	6 A NFC 10x38 gG AC, 400 V	Tube heating generator
S3	25 A NFC 10x38 gG AC, 400 V	Generator power supply unit

The fuse holders are located on the terminal strip on the left-hand side of the device.



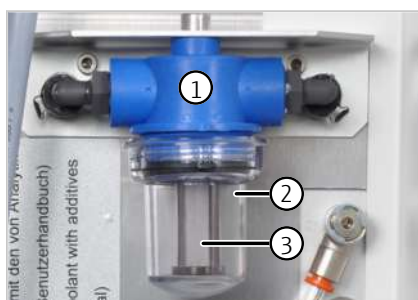
Figure 33 Device fuses

- ▶ Switch off the device via the power switch.
- ▶ Remove the cover from the side connections.
- ▶ Open the fuse holder toward the front.
- ▶ Replace the affected fuse.
- ▶ Close the fuse holder.
- ▶ Reattach the cover. Reattach the cover plate.
- ▶ Switch on the device via the power switch.

Inform customer service if the fault repeats itself.

7.2.10 Replacing the water filter

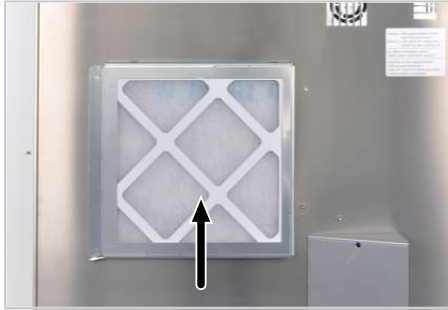
The water filter is located on the connector block on the left-hand side of the device. Check the filter cartridge in the filter monthly for contamination and clean the cartridge as required. Replace the cartridge at least once a year and if heavily contaminated. Only use filter cartridges supplied by Analytik Jena GmbH.



- ▶ Switch off the emission spectrometer and the recirculating chiller via the power switch.
- ▶ Put a bucket in place and unscrew the filter cup (2) clockwise from the water filter (1).
- ▶ Remove the filter cartridge (3) and rinse it under running water. Replace the cartridge if necessary.
- ▶ Refit the filter cartridge and cup.

7.2.11 Replacing the air filter

The air inlet filter is located on the rear of the device. Check the filter monthly and replace it if it is heavily contaminated.




- ▶ Pull the contaminated filter from the holder.
- ▶ Insert a new filter so that the arrow on the side of the filter point toward the device.

7.3 Sampler maintenance

7.3.1 Replacing the cannula and sample hose

The samplers are supplied with a cannula to which the sample hose is attached. The cannula and the sample hose are always replaced together.

- ▶ Switch off the sampler via the power switch.
- ▶ Disconnect the connection between the sample hoses of the sampler and the device.
- ▶ Carefully pull the sample hose from the hose guides on the automatic sampler.
- ▶ Unscrew the cannula from the holder on the automatic sampler. Remove the cannula with the sample hose and the connection pieces from the holder on the sampler.
- ▶ Prepare the new cannula with sample hose:
 - Fit the connection piece (1) onto the sample hose.
 - Push the conical nipple onto the cannula with the narrow side facing downward. Position the conical nipple close to the upper edge of the cannula.
 - Push the banjo bolt (3) onto the cannula from below. Screw the banjo bolt and the connection piece (1) to each other.
- ▶ Insert the cannula into the holder on the sampler. Fasten the cannula with the connection piece (4) in the holder from below. To do this, screw connection pieces (1) and (4) to each other.
- ▶ Guide the sample hose through the hose guides on the sampler (→ "Putting the ASPO 3300 into operation"  34).

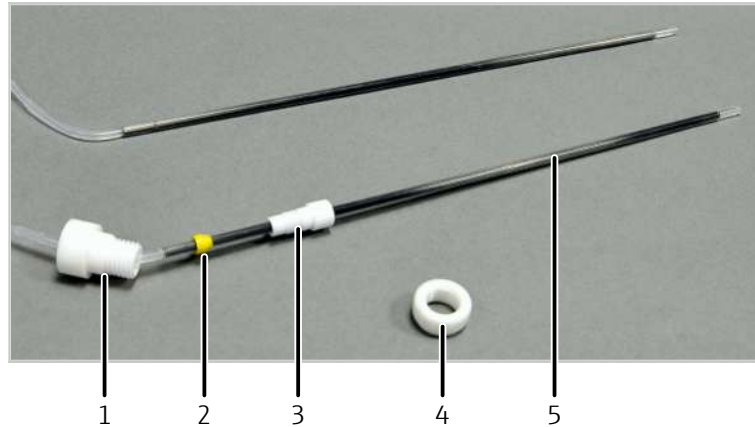



Figure 34 Replacing the cannula and sample hose of the sampler

- | | |
|---|---|
| 1 Connection piece (attachment to holder) | 2 Conical nipple |
| 3 Banjo bolt | 4 Connection piece (attachment to holder) |
| 5 Cannula with sample hose (single unit) | |

On older models, the cannula and the sample hose can be replaced individually.

- ▶ Switch off the sampler via the power switch.
- ▶ Disconnect the connection between the sample hoses of the sampler and the device.
- ▶ Carefully pull the sample hose from the hose guides on the automatic sampler.
- ▶ Unscrew the cannula from the holder on the automatic sampler.
- ▶ Unscrew the banjo bolt on both the cannula and the sample hose from the connection piece.
- ▶ Only use a straight-cut, round and unpinched hose end for the connection when replacing the sample hose.
- ▶ First push the banjo bolts and then one conical nipple with the conical side first onto the hose and the cannula.
The conical nipple and the hose or cannula end must be flush (see the figure below).
- ▶ Screw the banjo bolts into the connection piece hand-tight.
- ▶ Install the cannula in the sampler holder and guide the sample hose through the hose guides on the automatic sampler (→ "Putting the ASPQ 3300 into operation"  34).

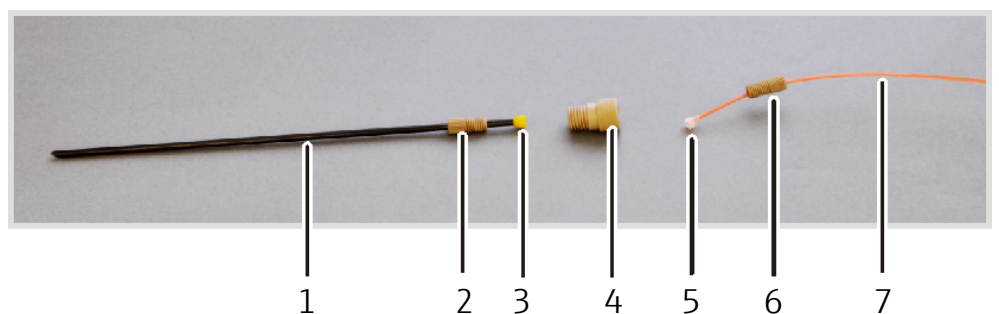


Figure 35 Cannula and sample hose of the sampler (disassembled)

- | | |
|------------------|--------------------|
| 1 Cannula | 2 Banjo bolt |
| 3 Conical nipple | 4 Connection piece |
| 5 Conical nipple | 6 Banjo bolt |
| 7 Sample hose | |

7.3.2 Replacing the pump hoses of the purging pump



CAUTION

Risk of chemical burns during hose replacement

Small quantities of acidic solutions can still be in the hoses.

- Wear protective gloves and clothing when replacing the hoses.
- Collect any leaking liquids with an absorbent sheet.

Replacing the hoses

- ▶ Switch off the sampler via the power switch.
- ▶ Place a flat container or absorbent material underneath the purging vessel.
- ▶ Release the clamping brackets on the pump and fold them down.
- ▶ Relieve the pump hoses and pull them out of the purging vessel connections.
- ▶ Pull the connection hoses for purging solution and waste from the pump hoses.
- ▶ Connect the pump hose for the purging solution to the lower intake connection (1a) of the purging vessel. Place the pump hose over the hose block from above and clamp it between two stoppers. Connect the intake hose for purging solution to the other end of the hose (1b). Immerse the intake hose in purging solution.
- ▶ Connect the pump hose for waste to the top outlet connection (2a) of the purging vessel. Place the pump hose over the hose block from below and clamp it between two stoppers. Connect the waste hose to the other end of the hose (2b). Insert the waste hose in the waste bottle.
NOTICE! Note the pump direction! The pump moves in a clockwise direction.
- ▶ Fasten the clamping bracket over the pump hoses via the clamping lever.
- ▶ Check the flow rate and adjust it either via the contact pressure or the pump speed.

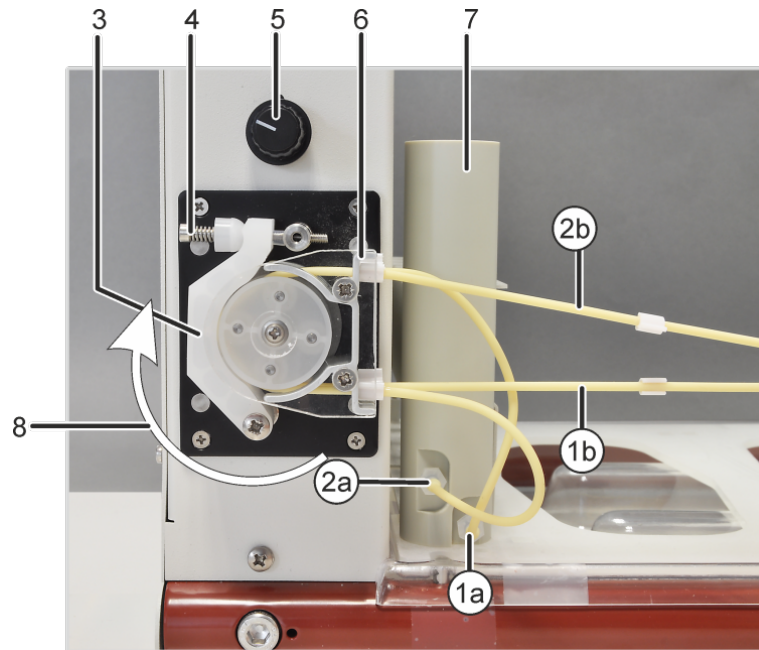


Figure 36 Purging vessel and pump on the sampler

- | | |
|---|--|
| 1a Intake connection for purging solution on the purging vessel | 1b Purging solution hose |
| 2a Waste connection on the purging vessel | 2b Hose to the waste container |
| 3 Clamping bracket | 4 Clamping lever with spring |
| 5 Pump speed controller | 6 Hose block for clamping the pump hoses |
| 7 Purging vessel | 8 Pump direction |

Setting the contact pressure and flow rate

The effective pressure on the hose is set via the clamping lever. To maximize both the service life of the hoses and the pump performance, set the contact pressure as follows:

- ▶ Loosen the screw on the clamping lever so that the clamping brackets no longer press on the hoses.
- ▶ Place the intake hose in purging solution. Insert the waste hose in the waste bottle.
- ▶ Switch on the basic device and the sampler via the power switch. Start the control software.
- ▶ Click the **[Autosampler]** button and switch to the **Function Tests** tab in the **Autosampler** window. Activate the **Wash pump** option and exit the window with **[OK]**.
- ▶ Tighten the screw on the clamping lever until the purging solution begins to flow. Tighten the screw by one further turn.
- ▶ Set the contact pressure on the pump hose for waste in the same manner.
- ▶ Adjust the flow rate of the pump via the rotary knob. The liquid level in the sampler must stay constant. Too much purging solution should not be allowed to spill over.
- ▶ Deactivate the **Wash pump** option in the **Autosampler** window.

7.3.3 Replacing fuses

Replace the fuses of the sampler as follows:

- ▶ Switch off the sampler via the power switch.
- ▶ Pull out the fuse holder. To do so, insert a screwdriver blade into the slot in the fuse holder and carefully pry out the holder.
- ▶ Replace defective power fuses. Only use T 5 A H 250 V, 5 x 20 mm type fuses.
- ▶ Insert the fuse into the clip marked with an arrow (see image).
- ▶ Connect the power cable and the serial connection (HOST) to the sampler.
- ▶ Switch on the sampler via the power switch.



Figure 37 Replacing the fuses on the sampler

7.4 Maintenance of the recirculating chiller: Replacing the cooling water



WARNING

Risk to health due to cooling water additives

The biocide used is corrosive and can cause an allergic reaction on skin contact.

- When handling the cooling water additive, wear protective glasses, clothing, and, in particular, gloves.
- Observe all notes and specifications in the safety data sheet.



NOTICE

Risk of device damage due to corrosion and algae growth

Only the use of the cooling water additive can effectively prevent damage due to corrosion or biological contamination of the device.

Damage to the device caused by operation of the device without cooling water additive is excluded from the warranty.


- Always add the cooling water additive (418-13-410-540) supplied by Analytik Jena GmbH to the cooling water.

The cooling water must be replaced at least once per year. The cooling water must always be replaced if the conductivity increases beyond 50 to 200 $\mu\text{S}/\text{cm}$.

- ⇒ Required tools: 10 L distilled/deionized water, cooling water additive set for recirculating chiller, suitable glass, plastic or stainless steel container for mixing the cooling water, bucket to collect the drained coolant
- ▶ Dissolve the contents of both bottles of the cooling water additive set (biocide and corrosion protection) on 10 L of water.
 - ▶ Start the wizard to replace the cooling water in the ASpect PQ control software. To do this, select the **Extras | Maintenance** menu item and click on the **[Change]** button.
 - ▶ Follow the instructions in the wizard:
 - Switch off the recirculating chiller.
 - On the recirculating chiller, disconnect the cooling water return flow connection and hold the hose in the collection vessel (bucket).
 - Switch the recirculating chiller back on and allow it to run until the flow of cooling water ends and only mist comes out.
 - Reconnect the hose to the cooling water return flow connection on the recirculating chiller.
 - Unscrew the sealing cap from the filling opening on the tank and insert the funnel.
 - Pour the coolant into the tank until the MAX level mark is reached.
 - Switch on the recirculating chiller and observe the level indicator. The level drops when the pump is operating.
 - Slowly continue to fill the tank with the coolant until the level stabilizes slightly below the MAX mark.
 - Remove the funnel and seal the filling opening with the screw cap.
 - Confirm the closing step in the wizard.
 - ▶ Wait for the message in the wizard indicating that the coolant has been replaced.
 - ▶ Exit the wizard.

8 Transport and storage

8.1 Preparing the device for transport

- ▶ Switch on the device and start the control software.
- ▶ Remove the cooling water from the system:
 - Start the wizard for replacing the cooling water in the control software.
 - Drain the cooling water and exit the wizard (→ "Maintenance of the recirculating chiller: Replacing the cooling water"  76).
- ▶ Switch off the device. Exit the control software and switch off the PC.
- ▶ Disassemble the torch, spray chamber and nebulizer and pack them.
- ▶ Remove the cover plate in front of the connections on the left rear side of the device.
- ▶ Disconnect all electrical connection cables from the device, the PC and the sampler.
- ▶ Disconnect all cooling water hoses from the device.
- ▶ Place an absorbent sheet underneath the connections to catch dripping liquid. Press the ring on the quick-release connector inwards and pull the hose from the connection for this.
- ▶ Disconnect the argon hose from the device. On the quick-release connector on the rear left side of the device, press the blue ring inwards and pull out the hose.
- ▶ Unplug the interface cables of the electrical components (sampler, PC) from the connections on the connection panel on the left rear side of the device.
- ▶ Reattach the cover plate in front of the connections on the left rear side of the device.
- ▶ Screw in the four transport handles as far as possible.
- ▶ Pack the device in the original packaging.

8.2 Moving the device in the laboratory



CAUTION

Risk of injury due to falling device

- Screw the four transport handles into the device as far as possible. Only this allows you to grip and carry the device safely.

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, four persons are required to transport the device, one person at each corner of the device.
- Firmly grip the device with both hands by the transport handles. 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.

8.3 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!

8.4 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.

8.5 Recommissioning the device

- ▶ Unscrew the handles and keep them in a safe place.
- ▶ Connect the exhaust hose to the smokestack of the emission spectrometer in a form-fitting manner.
- ▶ Remove the cover plate in front of the connections on the left rear side of the device.
- ▶ Install the gas supply:
- ▶ Connect the argon hose from the gas supply with the supplied T-piece. Insert the short hose pieces into the two argon connections as far as possible (→ "Supply and control connections"  17).
- ▶ If oxygen is used as an auxiliary gas: Insert the hose for oxygen in the connection.
- ▶ Connect the sampler and the PC with the device via the corresponding interfaces.
- ▶ Connect the device to power.
- ▶ Install the torch and the other glass components for the sample supply system (→ "Installing the sample supply system"  31).
- ▶ Install the recirculating chiller (→ "Installing the recirculating chiller"  80).
- ▶ Install the sampler and any other accessories (→ "Putting the ASPQ 3300 into operation"  34), (→ "Installing other accessories"  38).


- ▶ Reattach the cover plate in front of the connections on the left rear side of the device.
- ▶ Switch on the device and start the control software on the PC.

8.6 Installing the recirculating chiller



NOTICE

Risk of device damage due to incorrect operation of the recirculating chiller

- Observe the operating manual of the recirculating chiller.
 - Always use the Analytik Jena GmbH cooling water additive for the cooling water.
-
- ▶ Connect the device and the recirculating chiller with the cooling water hoses:
For easier distinction, one of the hoses is marked with hose clips at both ends.
 - Cooling water inlet flow connection on the recirculating chiller with the "In" connection on the device
 - Cooling water outlet flow connection on the recirculating chiller with the "Out" connection on the device
 - ▶ Connect the recirculating chiller to power and switch it on.
For the water-water cooler, install the cooling water circuit on the building side.
 - ▶ Prepare the cooling water (→ "Maintenance of the recirculating chiller: Replacing the cooling water"  76):
 - Start the ASpect PQ control software and start the wizard for replacing the cooling water.
 - Switch on the emission spectrometer.
 - Fill cooling water with the assistance of the wizard. Skip the part in the wizard on draining the cooling water.
 - ▶ Set the following parameters on the recirculating chiller:
 - Temperature: 18 °C
 - Set the cooling water pressure so that a water inlet flow of at least 1.5 to 2.0 l/min is achieved. The maximum pressure must not be exceeded here. Pressure (max.): 600 kPa (6 bar)

9 Disposal

Most waste produced via analysis is in the form of aqueous solutions. Aside from metal and heavy metal ions, these primarily contain various mineral acids used in sample preparation.

For safe disposal of this waste, all solutions must be neutralized with an alkaline solution such as a diluted sodium hydroxide solution. The neutralized waste must be disposed of correctly in accordance with statutory regulations.

Organic waste solutions must be disposed of separately and in accordance with statutory regulations.

At the end of its service life, the device and its electronic components must be disposed of as electronic waste in accordance with the applicable regulations.

10 Specifications

10.1 Technical data

10.1.1 Basic device technical data

PlasmaQuant 9100 Elite	Monochromator	Echelle grating double-monochromator with a focal length of F= 400 mm and variable gap; pre-monochromator with quartz prism, wavelength selection via additional reflected neon radiator
	Wavelength range	160 to 900 nm
	Wavelength accuracy	< 0.4 pm
	Spectral resolution	0.002 nm at 200 nm, 0.006 nm at 400 nm, 0.009 nm at 600 nm
	Experimental width at half height	≤ 3.5 ppm for As 193.696 nm, P 231.618 nm, Cd 228.022 nm
	Resolution	1:145 000
	Grating	Mechanically etched grating, 79 grooves/mm, blaze angle at 76°
	Photometer optical table enclosure	Modular optic design on a compact cast baseplate for stability and durability Protection against moisture, exhaust and chemical environmental influences
	Detector	Two-dimensional FFT backside illuminated CCD with high quantum efficiency and increased UV sensitivity
PlasmaQuant 9100	Monochromator	Echelle grating double-monochromator with a focal length of F= 400 mm and variable gap; pre-monochromator with quartz prism, wavelength selection via additional reflected neon radiator
	Wavelength range	160 to 900 nm
	Wavelength accuracy	< 0.4 pm
	Spectral resolution	0.006 nm at 200 nm
	Resolution	1:70 000
	Photometer optical table enclosure	Modular optic design on a compact cast baseplate for stability and durability Protection against moisture, exhaust and chemical environmental influences
	Detector	Two-dimensional FFT backside illuminated CCD with high quantum efficiency and increased UV sensitivity
Display types	Emission	Counts (ct)
	Intensity	Counts/second (ct/s)
	Concentration	5-digit value range (0.0001 to 99999), unit freely selectable
Signal evaluation	Spectral resolution	Spectra widths of 20 to 200 pixels

Analytical data	Sample type	Liquid	
	Type of nebulizer	Concentric nebulizer	
	Spray chamber	Cyclone chamber	
Power supply	Voltage	230 V \pm 10%	
	Frequency	50/60 Hz	
	Typical average power consumption	4500 VA	
	Maximum current consumption	32 A	
	Fuse protection (grid side)	32 A	
Device fuses	Fuse	Type	Protected circuit
	S1	10 A NFC 10x38 gG AC, 400 V	Spectrometer
	S2	6 A NFC 10x38 gG AC, 400 V	Tube heating generator
	S3	25 A NFC 10x38 gG AC, 400 V	Generator power supply unit
Safety circuits	Monitoring	<ul style="list-style-type: none"> ■ Plasma chamber door closing ■ Torch working position ■ Cooling ■ Argon supply ■ Plasma (optical monitoring) 	
Gas supply	Gas	Inlet pressure	Total consumption
	Argon \geq 4.6	600 kPa (6 bar)	13 to 21 l/min
	Permissible components: Oxygen \leq 3 ppm Nitrogen \leq 10 ppm Hydrocarbons \leq 0.5 ppm Moisture \leq 5 ppm		
	Oxygen \geq 4.5 (as optional auxiliary gas)	600 kPa (6 bar)	\leq 0.04 l/min
Ambient conditions	Temperature range	+15 °C to +35 °C, optimal is +20 °C to +25 °C, as constant as possible during measuring operations	
	Max. humidity	20 to 90 % at 20 °C	
	Air pressure	0.7 bar to 1.06 bar	
	Max. permissible altitude	2000 m	
	Storage	Temperature: -40 °C to +70 °C Use dessicant	
Dimensions and weight	Dimensions (W x H x D)	990 mm x 940 mm x 855 mm	
	Weight	170 kg	

10.1.2 Control computer technical data

Minimum requirements for the control computer	<p>PC with Windows 8.1 or Windows 10 (32/64 Bit)</p> <p>Graphics resolution 1280 x 1024 (1024 x 768 possibly with restrictions), Direct X 9, WDDM 2.0</p> <p>Processor: 1.6 GHz Dual Core CPU</p> <p>Working memory: 2 GB RAM (32 Bit), 4 GB RAM (64 Bit)</p> <p>Free disk space: 4 GB (SSD recommended)</p> <p>4 x USB 2.0 interfaces</p> <p>Mouse / trackball, keyboard</p> <p>A CD/DVD drive is required for installation.</p>
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10.1.3 Recirculating chiller technical data

Water-air cooler

Tank capacity	3.5 l
Dimensions (W x H x D)	460 mm x 703 mm x 735 mm
Supply voltage / frequency	110 V / 60 Hz 230 V / 50/60 Hz
Typical average power consumption	2900 VA
Cooling performance	3000 VA at 25 °C
Mass (empty)	92 kg
Silent Version (optional), noise level	≤ 57 dB
<ul style="list-style-type: none"> ■ Length of the water hoses ■ Length of the power cable 	<ul style="list-style-type: none"> ■ 3.5 m ■ 2.7 m
(For placement in adjacent rooms)	

Water-water cooler

Tank capacity	5 l
Dimensions (W x H x D)	360 mm x 590 mm x 470 mm
Supply voltage / frequency	230 V / 50 Hz
Typical average power consumption	160 VA
Cooling performance	3500 VA at 20 °C
Mass (empty)	33 kg
Noise level	≤ 50 dB
Max. water inlet flow temperature (primary side)	15 °C
Required water quantities	610 l/h (for 15 °C water temperature on the inlet side, 20 °C on the outlet side and $\Delta p = 40$ kPa)

10.1.4 ASPQ 3300 sampler technical data

Dimensions (W x H x D)	285 mm x 510 mm x 490 mm
Mass	15 kg
Supply voltage, frequency	100 to 240 V, 50/60 Hz
Fuse	T 5 A H 250 V, 5 x 20 mm
Typical average power consumption	75 VA
Racks	3 (sample vessels) 2 (special vessels)
Purging bottle	2 l

10.1.5 Technical data for other accessories

Sampler	Teledyne Cetac ASX-560	Dimensions (W x H x D)	580 mm x 620 mm x 550 mm
		Mass	12 kg
	Cetac Oils 7400	Dimensions (W x H x D)	570 mm x 490 mm x 540 mm
		Mass	23 kg
Dilution system	Teledyne Cetac SDX(HPLD)	Dimensions (W x H x D)	132 mm x 254 mm x 117 mm
		Mass	4.4 kg
	Accessories for quick sample supply	Cetac ASXPress Plus	Dimensions (W x H x D)
Switching valve			83 mm x 254 mm x 200 mm
Control unit			200 mm
Mass		1.3 kg	
Switching valve		1.4 kg	
Electrical connection data	The electrical connection data applies for all accessories listed.		
	Voltage	100 to 240 V (power supply unit)	
		24 V (accessory operating voltage)	
	Frequency	47 to 63 Hz	
Interfaces	USB		
	RS 232		

10.2 Guidelines and standards

Protection class and protection type	The device is protection class I. The housing is protection type IP 20.
Device safety	The device complies with the following safety standards <ul style="list-style-type: none">■ EN 61010-1■ ISO 9022-32-03-0
EMC compatibility	The device has been tested for radio interference elimination and interference immunity and fulfills the requirements of EN 61326-1.
Environmental compatibility	The device has been tested for environmental compatibility and meets the requirements according to <ul style="list-style-type: none">■ ISO 9022-2■ ISO 9022-3■ ISO 9022-32-03-0
EU directives	<p>The device meets the requirements of the directive 2011/65/EU.</p> <p>The device is designed and tested in accordance with standards meeting the requirements of EU directives 2014/35/EU and 2014/30/EU. The device leaves the factory in a sound condition with regard to technical safety. To maintain this condition and to ensure safe operation, the user must strictly observe the safety and operating instructions contained in this operating manual. For accessories delivered with the device and system components from other manufacturers, the information provided in their respective operating manuals has priority.</p>
Guidelines for China	The device contains substances subject to regulation (according to the directive GB/T 26572-2011). Analytik Jena guarantees that, if the device is used as intended, these substances will not leak within the next 25 years and therefore will not pose a threat to the environment or health within this time period.

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