

# **Operating Manual**

novAA 800 Atomic Absorption Spectrometer



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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 Notes on this user manual

The user manual describes the following models of the novAA product family:

- novAA 800 D Combined device for flame and graphite furnace techniques
- novAA 800 F for flame technique
- novAA 800 G for graphite furnace technique

In this manual all these devices are collectively called novAA 800. Any differences between the models are explained in the relevant section. Unless otherwise stated, the device shown on the illustrations is the combined device novAA 800 D.

The novAA 800 is intended for operation by qualified specialist personnel observing this user manual.

This user manual is addressed to personnel familiar with analysis and provides information about the design and function of the novAA 800 and the necessary know-how for the safe handling of the device and its components. Furthermore, the user manual includes information on the maintenance and servicing of the device as well as hints on potential causes for malfunctions and their correction.

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

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

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

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

Symbols and signal words

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



#### WARNING

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



#### CAUTION

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



#### NOTICE

Indicates potential damage to equipment or the environment.

# 1.2 Intended use

The novAA 800 is an atomic absorption spectrophotometer with deuterium background correction that can be used for the sequential determination of traces of metals and semi-metals in liquid or dissolved samples in routine analysis as well as for research purposes. Depending on the model, the device is equipped with a transversely heated graphite tube atomizer and/or a flame atomizer.

For the hydride technique and the HydrEA technique (used for coupling with the graphite furnace) there are hydride systems for batch and continuous operation.

The novAA 800 may only be used for atomic absorption spectrometry with the techniques described in this manual. Any departure from the instructions for proper use may lead to warranty restrictions and reduced manufacturer liability in the case of damage.

Not observing the safety instructions when handling the novAA 800 is considered noncompliant use deviating from the intended purpose. Safety instructions are to be found especially on the equipment itself, in section "Safety instructions" on page 9 and in the description of the relevant work steps.

# 2 Safety instructions

## 2.1 General notes

For your own safety and to ensure error-free and safe operation of the novAA800, please read this chapter carefully before using the appliance.

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

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

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

# 2.2 Safety labeling on the device

Warnings and information symbols have been attached to the novAA 800 which must always be observed.

Damaged or missing warnings and information symbols can cause incorrect actions leading to personal injury or material damage! Labels containing symbols must not be removed or moistened with methanol! Damaged symbol labels must be replaced with-out delay!



Fig. 1 Safety markings on the rear of the device

Rear of the device

Number	Warning / Information symbol	Meaning
		scope of application
1	25	The device contains restricted substances. Analytik Jena guarantees, that those hazardous substances may not leak out during the next 25 years when the device is used in accordance with its intended purpose.
2	Caution! Disconnect AC line before removing cover. Changing mains fuse only by authorized personnel.	Warning only for novAA 800 D + G Before opening the device hood switch off the device and disconnect the mains plug from the mains connection. The main inlet fuses (F1, F2) may only be re- placed by Analytic Jena customer service and au- thorized technical personnel.
3	Warning! Voltage on power point also by switched off AAS power switch! Pay close attention to the limit of the admissible current when connecting up individual components. Fuse also in N-Line!	(For meaning see warning text)
4		Before opening the device hood switch off the device and disconnect the mains plug from the mains connection.

Unlock power cable before opening!

Front and side panels of the device



Fig. 2 Safety markings on the front and side panels of the device

Number	Warning / Information symbol	Meaning scope of application
1		Hot surfaces! Risk of burns at the hot graphite furnace and burner! (Position: at the height ad- justment)
2		Read the operating manual before commencing work. (Position: at the mains switch on the right- side of novAA 800)
3	Caution! Hot surface! Caution! Emission of UV radiation!	Hot surfaces! Risk of burns at the hot graphite furnace and burner! Dangerous UV radiation! Do not look into the lamp beam, into the flame or the graphite furnace without UV protection goggles.
	Danger of short circuit! Handling with jewels not allowed!	Short circuit warning only for novAA 800 D + G Do not wear any metallic jewelry (especially around the neck). Not observing this instruction may cause a short circuit in the electrically heated furnace. The jewelry may become very hot and cause burns.
4	Warning! Switch off lamps when door is opened!	The ultraviolet light emitted by the (deuterium) hollow cathode lamps can cause eye and skin damage. Turn off the lamp current via ASpect LS software before opening the lamp chamber.

### 2.3 Requirements for the operating personnel

The novAA 800 must only be operated by qualified specialist personnel instructed in the use of the device. The instruction must also include conveying the content of this user manual and the user manuals of other system components.

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

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

# 2.4 Safety instructions, transport and installation

Observe the following notes:

 The novAA 800 is always installed by the service department of Analytik Jena or by specialist personnel that was authorized and trained by Analytik Jena. Independent assembly and installation are not permitted. Incorrect installation can create serious hazards.

- The various different models of the novAA 800 product family weigh between 95 and 130 kg. Use a lift truck to transport the device.
- Four people are required to move the device in the laboratory by holding the device on four firmly screwed-in carrying handles.
- Perform a professional and documented decontamination of the device before returning it to Analytik Jena. The decontamination protocol is provided by the service when the return is registered. Analytik Jena must refuse acceptance of contaminated devices. The sender may be liable for any damage caused by inadequate decontamination of the device.

Protection against explosion and fire

- The novAA 800 must not be operated in an explosive environment.
  - Smoking and open flames in the operating room of the novAA 800 are prohibited!
  - The operator is responsible for establishing a control method to ensure that the NO<sub>2</sub> and acetylene connectors are leak-tight.

## 2.5 Safety instructions for operation

- Prior to starting up the device, the operator of the novAA 800 must verify the proper condition of the device including the condition of its safety equipment. This applies in particular after each modification or extension of the device or its repair.
- The device must only be operated if all protective equipment (e.g. covers and doors) 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, the operator must have free access to all connections and the power switch on the right side of the device as well as to the power strip.
- The ventilation equipment on the device must be in good working condition. Covered ventilation grilles or slits etc. may cause the device to break down or damage the device. When placing the device and system components observe a minimum clearance of 150 mm to walls and neighboring installations.
- Prevent any liquids from entering the inside of the instrument. The liquids might get into contact with electronic components and cause a short circuit.

#### 2.5.1 Safety instructions for electrical equipment

Work on the electrical components of the novAA 800 may only be performed by a qualified electrical technician according to applicable electro-technical regulations. Lethal voltages may occur in the device! Contact with live components may cause death, serious injury or painful electrical shock.

Observe the following notes:

- The mains plug must be connected to a proper CEE power socket 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 type plate of the equipment. The protective effect must not be invalidated by the use of an extension line which does not have a protective conductor.
- The novAA 800 and its system components must always be switched off before being connected with one another.
- The auxiliary components that are to communicate with each other, for example PC, monitor, printer, hydride system and cooling unit, have to be connected to the power strip that is supplied with the device. The compressor needs a separate power supply. When connecting own components to the power strip, observe the maximum allowable drain current (see section "Energy supply" on page 40).
- Before opening the device it must be switched off on the device switch and the mains connector must be disconnected from the mains outlet!
- The novAA 800 must be switched off before carrying out any electrical work and the mains plug must be pulled out. Safe disconnection from the mains can only be achieved by pulling out the mains plug. The power strip is still energized, even when the novAA 800 is switched off at the mains switch on the right side wall. The power strip socket to which the novAA 800 is connected is protected by a fuse in both wires, one in the L conductor (line) and one in the N conductor (neutral). This can mean in the case of a fault that connected components are supplied with voltage via the L-line, but no current can flow through the N-line, i.e., without a more thorough check, the connected devices appear to be voltage-free, which is not true.
- Any work on the electronics (behind the device enclosure) may only be carried out by the customer service of Analytik Jena and specially authorized technicians.

#### 2.5.2 Hazards associated with the operation of the flame and the graphite furnace

HCL, D<sub>2</sub>-HCL, the heated graphite tube (T > 1000 °C) and the flame of the burner transmit optical radiation (in the UV range and the visible range). Do not look into the rays emitted by the lamp, the graphite tube or the flame without UV protection goggles. Protect the skin against UV radiation.

Switch off the lamp by means of the control and analysis software ASpect LS before opening the lamp door: Set the lamp current in [mA] to zero in the OPTICAL PARAMETERS section of the SPECTROMETER / CONTROL window. Open the drop-down list BACKGROUND CORRECTION and select the option NO BACKGROUND. Click [CONFIGURE]. Negate the error message.

To observe the placement of the samples or the drying of liquid samples, the dental mirror may only be inserted into the beam path from the left side of the graphite furnace. When observing on the right side of the furnace, UV radiation may be reflected.

- The sample chamber door (safety glass pane) must be closed and the flame supervised when it is burning. Ensure that the flame detector is working correctly.
- For devices with hydride technique, only work with the sample chamber door (safety glass pane) closed.

- The fuel gas pressure must not fall below 70 kPa to prevent the flame from firing back. The integrated pressure sensor will automatically switch off the novAA 800 if this condition is not met. In addition to that, monitor the pressure on the pressure gage of the gas supply.
- When using graphite furnace technique, do not look into the graphite furnace opening without wearing protective goggles. Sputtering sample substances and hot graphite particles may cause eye and face injuries.
- High temperatures occur during flame and graphite furnace operation. Do not touch hot parts such as the burner head or the graphite furnace during or immediately after a measurement. Observe the required cooling times.
- Do not wear any metallic jewelry (especially around the neck) when working with the novAA 800 D and G. Not observing this instruction may cause a short circuit in the electrically heated furnace. In case of a short circuit, the jewelry may become very hot and cause burns.
- Electromagnetic dispersion fields with flux densities ≤ 100 µT occur in the vicinity of the sample chamber due to the heating of the graphite tube.
- When using graphite furnace technique, the sound level may rise up to 55 dBA. If the nitrous-oxide-acetylene flame blows back into the mixing chamber, the momentary sound level lies below 130 dBA.

#### 2.5.3 Safety instructions relating to the formation of ozone and toxic vapors

The UV radiation of the hollow cathode lamps (HCL,  $D_2$ -HCL) and the  $N_2O$ /acetylene flame causes an interaction with the surrounding air to form toxic concentrations of ozone exceeding the permissible limit. Furthermore, toxic byproducts may escape from the samples or while the samples are processed.

Observe the following note:

- The novAA 800 may only be operated when the exhaust unit is activated.
- Always keep the sample chamber closed when the flame is lit.

#### 2.5.4 Safety instructions for compressed gas cylinders and systems

Observe the following notes:

- The operating gases (argon, acetylene and nitrous oxide) are taken from compressed gas containers or local compressed gas systems. The required purity of the gases must be ensured.
- Pure oxygen or oxygen-enriched air must not be used as an oxidant when using flame technique. There is a risk of explosion.
- Work on compressed gas cylinders and systems must only be carried out by individuals with specialist knowledge and experience in compressed gas systems.
- The safety instructions and guidelines for operating compressed gas cylinders or compressed gas systems that apply at the operating location must be strictly complied with.
- Compressed air hoses and pressure reducers may only be used for the assigned gases.

- Incoming piping, screwed joints and pressure reducers for nitrous oxide (N<sub>2</sub>O) must be kept free of grease.
- Pay particular attention to escaping acetylene! Combined with air, acetylene forms highly flammable mixtures. The gas can be identified by its distinct garlic-like odor.
- Operate the acetylene cylinder only in an upright position and secured against falling over. When the cylinder pressure is lower than 100 kPa, the acetylene cylinder must be replaced to avoid acetone entering the automatic gas control.
- The operator must carry out weekly safety checks regarding the status and for leaks on all gas supplies and connectors including the ones on the device itself. Possible pressure losses from closed systems and lines under pressure are to be determined. Leaks and damaged must be repaired without delay.
- The gas supply must be closed prior to inspections, service and repairs!
- After successful repair and service of the components of the compressed air cylinders or system the device must be checked for sound operation prior to recommissioning!
- Unauthorized assembly and installation are not permitted!
- After changing the gas cylinder, thoroughly ventilate the cylinder location.

#### 2.5.5 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.

Observe the following notes:

- When handling dangerous substances local safety codes and guidelines must be observed.
- Warnings on the labels must always be observed. Only use labeled tubes. Use suitable body protection (laboratory coat, safety goggles and rubber gloves) when handling samples.
- The novAA 800 must only be operated under an activated laboratory exhaust hood (hazards arising from the formation of ozone, combustion gases produced by the samples, poisonous and combustible by-products from sample preparation processes).
- Keep flammable and explosive substances away from the flame.
- 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.
- Sodium borohydride (NaBH<sub>4</sub>) is highly corrosive, hygroscopic and, in solution, extremely aggressive. Avoid dripping and spilling of reduction agent.
- Biological samples must be handled according to local guidelines regarding the handling of infectious material.
- When measuring material containing cyanide you have to make sure that prussic acid cannot be generated in the waste bottle, i.e. the waste solution must not be acidic.

Examples of organic

solvents

- Ensure that all residue liquid from the nebulizer and the automatic sampler is directed into the waste bottle supplied.
- The operator is responsible for ensuring that waste materials such as drained coolant and residue liquid from the waste bottle are disposed of in an environmentally responsible manner and according to local regulations.

Methyl isobutyl ketone (MIBK)	Flammable, highly volatile, noxious-smelling
Toluene	Flammable, hazardous to health
Kerosene	Flammable, hazardous to the aquatic environment, hazardous to health
Methanol, ethanol, propanol	Flammable, partly acutely toxic
Tetrahydrofuran (THF)	flammable, hazardous to health, extremely volatile, dissolves polyethylene and polystyrene

This list is not exhaustive. Other solvents could be considered when using the novAA 800. If in doubt about the potential hazards ask the manufacturer to provide more detailed information.

#### 2.5.6 Decontamination in case of biological contamination

Observe the following notes:

- The operator is responsible for carrying out suitable decontamination should the device be contaminated externally or internally with dangerous substances.
- Spots, drops or larger spillages should be removed and cleaned using an absorbent material such as cotton wool, laboratory wipes or cellulose. Then, wipe the affected area with a suitable disinfectant such as Incidin Plus solution.
- 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 novAA 800 must not be moistened with methanol.

### 2.6 Behavior during emergencies

Observe the following notes:

- If there is no immediate risk of injury, in hazardous situations or in case of accidents, immediately switch off the novAA 800 using the mains contactor on the right side wall of the device. Disconnect the mains plug from the mains socket.
- Make sure that the mains plug is readily accessible.
- Switch off the installed components using the mains switch of the connected power strip. Ensure that the positioning of the power strip allows quick access.

Caution! For the PC there is a risk of data loss and damage to the operating system!

• Close the gas supply immediately after switching off the devices, if possible.

# 2.7 Safety instructions: service and repair

Observe the following notes:

- The novAA 800 is categorically unpacked and assembled by the customer service department of Analytik Jena or its authorized and trained specialist personnel. Unauthorized servicing can lead to maladjustment or damage of the device. Therefore, the tasks that the operator is allowed to carry out are strictly limited to the tasks listed in chapter "Service and maintenance" on page 84.
- The exterior of the novAA 800 must only be cleaned with a damp not dripping cloth. Use only water and, if required, customary surfactants.
- For cleaning the sample compartment and transport channels (tube system) of the novAA 800 the operator is required to establish appropriate safety precautions – particularly in terms of contaminated and infectious materials.
- If water or other liquids are found to leak out of the instrument, contact the Analytik Jena service engineers.
- 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.

# 3 Function and setup

# 3.1 AAS techniques

The following atomization techniques are available for the devices of the novAA 800 product family:

Atomization technique	novAA 800 F	novAA 800 G	novAA 800 D
Burner-nebulizer system (flame technique)	$\checkmark$	-	$\checkmark$
Transversely heated graphite tube (graphite furnace tech- nique)	-	$\checkmark$	$\checkmark$
Cell unit (hydride and mercury cold vapor technique)	$\checkmark$	$\checkmark$	$\checkmark$
Transversely heated graphite tube with Ir/Au coating (HydrEA technique)	-	$\checkmark$	$\checkmark$



Fig. 3 Sample chamber of the novAA 800 D

In the combined device novAA 800 D the flame atomizer and the graphite furnace are both attached to a bracket on the height adjustment that can be tilted by 60°. Tilting the bracket forwards into locking position puts the graphite furnace into the optical axis. Tilting the bracket backwards up to an adjustable stop puts the flame unit into the optical axis. Since both atomizers are aligned with the axis, changing from one atomization techniques to the next can be done in a few minutes. The control and analysis software ASpect LS uses the height adjustment to move the atomizer into the correct operating position automatically. The depth of the atomizer within the sample chamber is factory preset and may be adjusted manually for the flame atomizer using an adjustment screw.

The novAA 800 F (flame) and the novAA 800 G (graphite) are both equipped with one atomizer.



Fig. 4 Sample chamber of the novAA 800 F

The core element of the graphite furnace mode is a closed, transversely heated graphite furnace into which the samples are loaded from the top.

For flame operation, the novAA 800 is designed as a double-beam instrument, which can also be used in single-beam operation. Core piece of the flame mode is the mix-ing-chamber-nebulizer system with direction-independent stable nebulizer.

The time-controlled infection module SFS 6 is available for the flame injection technique. It switches the valves in a constant stream of carrier solution to couple the sample segments.

The hydride technique with latest-generation hydride systems (HS 50, HS 55 modular, HS 60 modular) is the preferred process for the detection-sensitive determination of the hydride-forming elements As, Bi, Sb, Se, Sn, Te and Hg. The cell unit of the hydride system is placed on the mixing chamber instead of the burner in the novAA 800 D and F and on the clamped connector in the novAA 800 G.

Alternatively, the combined device novAA 800 D and the novAA 800 G allow coupling hydride technique with graphite furnace technique. The HydrEA technique ("Hydride technique with electro-thermal atomization") consists in accumulating metal hydrides or mercury vapor on the iridium-coated or gold-coated graphite tube and atomizing them at temperatures of 2100 °C (metal hydrides) or 800 °C (mercury). This allows attaining a very high sensitivity.

#### 3.2 Optical principle

The novAA 800 is a double-beam instrument, which can be used in single or double beam mode, depending on the selected technique. On the left side the 8-lamp turret (item 8 in Fig. 5) is vertically arranged. The lamp turret can be equipped with

1.5" hollow cathode lamps (HCL) as the primary radiation source. In front of the lamp turret there also is a deuterium hollow cathode lamp ( $D_2$ -HCL) (item 7 in Fig. 5) in vertical arrangement for the classical background compensation.

An optical beam splitter (item 9 in Fig. 5) with reflection and transmission fields in chess-board pattern unites the radiation of the active primary HCL with the continuum radiation of the  $D_2$ -HCL and splits it simultaneously into the sample and reference beams. Identical beam paths with the same beam distribution and density in the spatial angle used for both radiation sources ensure the optimal background compensation with the  $D_2$ -HCL.

The reference beam is routed behind the sample chamber. A rotating sector mirror (item 5 in Fig. 5) with 90° reflection sector and transmission sector brings the sample and reference beams together.

For the graphite furnace technique with  $D_2$  background correction, the novAA 800 is operated as a single-beam instrument.



Fig. 5 Optical diagram of the novAA 800

- 1 Monochromator mirror
- 2 Mesh
- 3 Slit aperture
- 4 Si-hybrid receiver
- 5 Sector mirror
- 6 Atomizer: graphite furnace or burner-nebulizer system

- 7 Deuterium hollow cathode lamp (( $D_2$ -HCL)
- 8 Lamp turret with 8 hollow cathode lamps
- 9 Beam splitter mirror

The sample beam or combined sample/reference beam is projected onto the entrance slit of a mesh monochromator (items 1 and 2 in Fig. 5). The mesh monochromator is equipped with fixed slits of 0.2 nm / 0.3 nm / 0.5 nm / 0.8 nm / 1.2 nm bandwidth. It

selects the resonance wavelengths assigned to the element. The wavelength of the monochromator is set according to the theoretical number of iterations, based on the zero order as an initialization point and corrected by an amount which is calculated from the device-specific wavelength interpolated function which is available as a polygon curve. 9 interpolation points are distributed equally over the wavelength range from the zero-th order up to 900 nm.

A peak-pick program is used to find the maximum of the particular line. The wavelength is set via a step-motor-driven wavelength drive with a resolution of 0.005 nm per step.

A Silicon-hybrid receiver (item 4 in Fig. 5) at the exit of the monochromator measures the intensity of the impinging radiation synchronously with the clocking of the light sources.

The optional accessory Air Purge Kit (APK) can be used to purge the spectrometer with purified compressed air. The use of this equipment is advisable when the novAA 800 is used in an operating environment with large quantities of dust, such as a mine.

#### 3.3 Measurement principle

The element-specific absorption of the radiation of a hollow cathode lamp is measured by atoms in the base state. In this, the absorption signal is a measure for the concentration of the relevant element in the analyzed sample. The HCL delivers a line spectrum from which a suitable resonance line is decoupled by the monochromator.

The continuum radiation of the D<sub>2</sub>-HCL is used for compensating the background absorption. The radiation of the line radiator (primary HCL) with its very narrow base line (resonance line) is element-specific and weakened non-specifically by scattering. In doing this, the total radiation is recorded. The radiation of the D<sub>2</sub>-HCL is mainly weakened by the broad band, non-element-specific absorption, the minimum element-specific part can be neglected. The formation of the difference between the two signals gives the element-specific absorption.

In flame mode, the novAA 800 can be used both as a single-beam and as a doublebeam instrument. For the hydride technique the unit is used as a single-beam device because a zero calibration is performed immediately before the integration period. For the flame technique, double-beam operation is preferred for on-the-spot measurements in the integration modes "Mean value" or "Moving average" if it is not possible to wait until the end of the warm-up time of the lamps.

#### 3.4 Lamp turrets and lamps

The novAA 800 has an 8-lamp turret with a write/read unit for coded lamps. The coded lamps are equipped with transponders. The following information is saved: lamp type, element(s), serial number, maximum/recommended lamp current and operating hours. The use of uncoded lamps is possible. The lamp turret is designed for hollow cathode lamps with a standard bulb diameter of 37.1 mm. The individual lamps are rotated (PC-controlled) into the beam path, switched on and adjusted according to the pitch circle in steps of 0.1 mm. A second heat circuit ensures that a second HCL can be preheated at the same time.

The continuum radiator, a deuterium hollow cathode lamp ( $D_2$ -HCL), is installed in a separate bracket (item 2 in Fig. 31 on page 54).



Fig. 6 Lamp turret with reader

- 1 Reader for coded chips
- 2 Lamp with coded chip (transponder)
- 3 Carrier plate for 8 lamps

### 3.5 Electrothermal atomizer

The electrothermal atomizer (EA) is an integral part of the novAA variants novAA 800 D and G and a core element for working in EA mode and with the HydrEA technique.

The furnace system is equipped with a graphite tube that is heated by contact elements positioned transversely to the coat of the tube. The transversely heated graphite tube serves as an atomizer for the liquid sample injected with the AS-GF autosampler. The required temperature of the graphite tube in the furnace is regulated by means of a microprocessor controlled Ohmic heating.

Characteristics of the graphite furnace

- Constant temperature ratios along the entire tube length
- Realization of linear temperature-time runs according to a sensorless control model on the basis of saved thermoelectrical parameters and an adaptive control
- Protective gas flows, independent of each other and symmetrical to the furnace center, which ensure effective graphite tube and furnace window cleaning, and

which also ensure fast and safe transport of the thermally disintegrated products of the sample for disposal

 Low consumption of protective gas while ensuring effective protection against the interference with atmospheric oxygen.

When combined with the deuterium background compensation, the graphite furnace technique achieves high levels of selectivity and sensitivity allowing the determination of traces and ultra-traces even in samples with a complex matrix.



Fig. 7 Graphite furnace in the sample chamber

- 1 Cooling water connections: red hoses
- 2 Furnace window
- 3 Radiation sensor
- 4 Gas connections: white and black hoses

- Sensor connection for cooling water temperature
- 6 Power cable
- 7 Fuse on the graphite furnace
- 8 Dosing opening with graphite funnel
- 9 Furnace clamps with electrodes

During analysis, each sample has to pass one furnace program (temperature-time program). The furnace program consists of four basic steps:

5

- Drying the sample
- Thermal pretreatment, separation (ashing or pyrolysis) of distorting incidental sample substances (matrix)
- Atomizing the sample
- Cleaning the graphite tube by baking out and preparing for the next measurement

The operator has the option to optimize these basic steps for each analysis problem with the ASpect LS control software.

A safety circuit prevents the graphite furnace in the novAA 800 from continued and uncontrolled heating in case of a communication failure between the control (PC) and the AAS. The temperature sensor is attached to the rear of the stationary furnace

(item 5 in Fig. 7). The safety circuit disconnects the main power supply of the device if the cooling water temperature reaches  $\geq 100$  °C.

This prevents damage to the device resulting from continued heating of the furnace. Once the cooling water temperature has fallen below the shutdown temperature, the novAA 800 can be switched back on and re-initialized.

#### 3.5.1 Graphite tube furnace

The transversely heated graphite tube is pneumatically pushed against circular electrodes and held in this position. The electrodes are installed in two water-cooled metal bodies, the stationary and the movable part of the furnace. There is another graphite component located between the metal bodies that support the electrodes, the furnace shroud. Together with the electrodes it forms an enclosure around the graphite tube, which stabilizes the thermal radiation conditions of the graphite tube and also guarantees chemically inert conditions. The graphite tube is pre-adjusted by means of defined support points in the furnace while the atomizer is open. When the movable part of the furnace is closed, the tube is lifted to its final position in a reproducible movement and pressed into the contacts, without coming into contact with the furnace shroud.



Fig. 8 Graphite furnace, opened

- 1 Furnace window
- 2 Stationary part of the furnace
- 3 Graphite tube, inserted
- 4 Dosing opening with graphite funnel
- 5 Furnace shroud

- 6 Furnace window
- 7 Movable part of the furnace, opened
- 8 Seal of the water channel

When changing from the wall-type tube to the platform tube, bear in mind that these special graphite tubes cover the free opening for the beam passage on one side. When selecting the respective technique, the motor-driven height adjustment moves to the optimum height position in a software-controlled movement.

#### 3.5.2 Gas flows in the furnace shroud

The furnace shroud houses the gas channels for the separate supply of the primary gas flow (purge gas) and the outer gas flow (protective gas). It is possible to add oxidizing and reducing gases to the mixture of the inner gas flow to support the pyrolysis. Avoid temperatures > 500 °C when using air to prevent the graphite tube from corroding.

The purpose of the inner gas flow is to remove all gases that are produced during the drying process and the pyrolysis from the graphite tube.

Furthermore, the inner gas flow prevents analytes from condensing on the furnace window and has an influence on the dwell time of the analyte atoms within the beam path. The atomization usually interrupts the inner gas flow to allow the atoms to dwell as long as possible within the beam path of the graphite tube. The desired result is a high sensitivity.

The outer gas flow sweeps the graphite tube and is directed through the funnel insert to the outside. The outer gas flow permanently surrounds the graphite tube with inert gas to protect it from oxidizing with the atmospheric oxygen.



Fig. 9 Inner and outer gas flows in the graphite furnace

1, 3 Inner gas flow (purge gas)

2 Outer gas flow (protective gas)

A cylindrical joint to the stationary part of the furnace is used to distribute the heat in the furnace shroud and for dissipating the heat. This allows heating up the interior walls of the atomizer to such high temperatures that prevent the analyte (the sample) from condensing.

The cone attachment on the opposite side of the furnace shroud forms a precisely defined gap in the rotatable part of the furnace together with the insulating ring ensuring that the cell interior is sealed against the ingress of ambient air. In the event of a tube rupture in the furnace shroud, the insulating ring in the movable furnace part prevents a short circuit between the furnace parts.

The furnace shroud is drilled through in the direction of the optical axis, the outer cylinders support the furnace windows (quartz cell windows). For cleaning, the windows can be pulled off with a twisting motion.



Fig. 10 Graphite furnace shroud

# 3.5.3 Graphite tube variations, furnace parts and inserts

There are two graphite tube models: the standard graphite tube (wall-type tube) and the graphite tube with PIN platform.



Standard graphite tube

Fig. 11 Variants of the graphite tube



1, 4 Cylinder for furnace windows 2, 3 Support: cone attachment

Graphite tube with PIN platform

Graphite tube model	Applicable total volume	Use
Standard graphite tube	max. 50 μL	Aqueous samples (samples not requiring complex analysis)
Graphite tube with PIN platform	max. 40 μL	Aqueous samples (samples requiring complex analy- sis)



Fig. 12 Furnace shroud, adapters and inserts

No.	Furnace Part / Insert	Function
1	Pipetting insert	Funnel opening to the pipetting channel
2	Adjusting aid	Adjusting the autosampler AS-GF
3	Electrode (2 per furnace)	Electrical contact to the tube wing
4	Furnace shroud	Receptacle for the graphite tube

#### 3.5.4 Radiation sensor

The radiation sensor is located on the right side of the graphite tube furnace and is inclined in relation to the direction of radiation. It recalibrates the tube temperatures by receiving radiation from the interior of the graphite tube on a sandwich receiver. Using two wavelengths for detection, an independent quotient signal is derived for temperature measurement which is independent of the degree of radiation of the graphite tube. Recalibration takes place when formatting the graphite tube.

#### 3.5.5 Furnace camera

The furnace camera is an optional accessory that can be switched on via the software control. The image recorded by the furnace camera would then be displayed in a separate window on the ASpect LS user interface. The furnace camera monitors the process, beginning with the injection of the sample into the graphite tube through to completion of the drying process. This allows the operator to control and, if necessary, correct the immersion of the dosing tube into the graphite tube, the dispensing of the sample and other components as well as the drying procedure. The furnace camera automatically shuts down before a pyrolysis process. The camera allows looking into the graphite tube from the left. The interior is illuminated by a LED from the right.

#### 3.6 Accessories for the graphite tube technique

#### 3.6.1 Autosampler AS-GF

In the graphite technique the autosampler AS GF is used to introduce liquid samples. When using the HydrEA technique, it deflects the reaction gas into the graphite tube. Manual pipetting is not recommended because of the poor reproducibility rate. The autosampler AS-GF accepts defined volumes of different solutions and places them into the graphite tube. It enables the

- Addition of up to five modifiers to the sample solution
- Transport of the sample solution to the thermal pretreatment in the tube
- Enrichment of samples
- Placement of components in the preheated tube
- Separate transport of components with intermediate purging
- Automatic preparation of standards by dilution or by different volumes
- Fixed, preselected or intelligent sample dilution
- Fully automatic multi-element mode (night mode possible)

The sample tray of the AS-GF provides enough space for 100 sample cups (with V = 1.5 mL) and 8 central cups for diluent, special samples, standards, modifiers etc. (with V = 5 mL).



- 1 Autosampler arm with cannula locking system
- 2 Tube guide with dosing tube
- Sample tray with 3
- sample tray cover 4 Metering syringe (500 µL)
- Waste bottle
- 5
- 6 Storage bottle for purge solution (or diluent)

Autosampler AS-GF Fig. 13

The AS-GF is attached to the respective receptacles provided in the sample chamber and electrically connected to the novAA 800. The device parameters of the AS-GF can be set with the ASpect LS control software.

#### Mobile cooling unit KM 5 3.6.2

The graphite tube furnace of the novAA 800 is cooled by the mobile cooling unit KM 5 via a circulating cooling system. The working principle is an air-cooled heat exchanger with fan. The cooling water temperature is preset to 35 °C. The cooling unit can only

cool effectively, if the preset value is at least 7  $^\circ C$  above room temperature. The maximum set point value is 50  $^\circ C$ .

The KM 5 has to be filled with 5 L of softened water (not distilled water). For installation and initial start-up of the device, please observe the information provided in the separate operating instructions of the mobile cooling unit KM 5.

#### 3.7 Flame system

Flame atomic absorption spectroscopy is used for determining trace elements in the concentration range from mg/L to  $\mu$ g/L and for determining the basic composition of the major components. It requires a flame with constant properties. Furthermore, the flame composition must be compatible with the element to be analyzed.

Motorized vertical adjustment of the nebulizer-mixing-chamber-burner system by 14 mm makes it possible to move the flame zone with the maximum absorption into the direction of the beam.

The sample solution is aspirated by a pneumatic nebulizer and sprayed into the mixing chamber. In the mixing chamber the sample aerosol is mixed with acetylene and oxidant before it emerges from the burner slot. The flame is either 5 or 10 cm long and a few millimeters wide, depending on the burner used. It is irradiated over its full length. For the measurement of main components, the burner can be rotated by max. 90° on the mixing chamber tube (transverse position). This reduces the absorption path. Correspondingly, the sensitivity is lower. It is possible to set the rotation of the burner using the scale on the burner.

#### 3.7.1 Automatic gas control

The automatic gas control ensures that the flame is fed with a defined flow of acetylene and oxidant at a constant non-fluctuating pressure level. This guarantees the safe and hazard-free ignition and quenching of the flame. The automatic gas control has three gas inlets for acetylene, air and nitrous oxide.

A proportional control valve sets the fuel gas flow in steps of 5 L in a range between 40 and 315 NL/h of acetylene. The airstream first fills a reservoir with a capacity of 500 cm<sup>3</sup> before the air flows to the nebulizer. The air in the reservoir is used to quench the flame in regular operation and in the event of an accident. The oxidant flow to the nebulizer is calculated from the set configuration and the inlet pressure. The oxidant flow is measured and monitored. If auxiliary oxidant is used, the auxiliary oxidant flow (air/nitrous oxide) is regulated in three stages.

A filament ignites the flame. The filament is swung from the rear panel of the sample chamber to the center of the burner. It is possible to switch from the acetylene-air flame to the acetylene-nitrous-oxide flame by blocking the air supply and adding nitrous oxide. The acetylene flow is increased at the same time. The acetylene-nitrous-oxide flame is quenched in reverse order. The switching of the flames is executed in a fully automated process controlled by the ASpect LS software.

#### 3.7.2 Burner-nebulizer system

The nebulizer uses the sample solution to generate the aerosol required for the atomization in the flame. The oxidant passes the lateral connection to enter the nebulizer and flows through the ring-shaped slit formed by the corrosion-proof platinumrhodium alloy cannula and the PEEK nozzle. The resulting negative pressure pulls the sample solution out of the cannula and aspirates more sample solution. The aspiration rate and the fineness of the aerosol are determined by the relative position of the cannula to the nozzle. It can be set manually with an adjustment screw and a lock nut.

The resulting sample aerosol strikes the baffle ball. Larger droplets condense on the baffle ball and run off via the siphon. The fuel flow strikes the surface of the baffle ball at a right angle. The aerosol that is produced passes the mixing chamber to flow to the burner. On the way through the mixing chamber, an equilibrium is reached. Other large droplets are separated by gravity and run off via the siphon. The aerosol is dried in the flame. During this process, the size of the droplets must be small. Fast evaporation of drops when entering the flame is a precondition for atomizing the sample in the hot zone of the flame. An incomplete vaporization of the extracting agent has a negative effect on the accuracy of the analysis result. At the same time, the unvaporized droplets disperse the radiation which results in an increased background absorption.

The mixing-chamber-nebulizer system is designed to allow the formation of a very fine aerosol from the aspirated samples. The system requires little maintenance because the siphon is located directly next to the nebulizer. Large drops drain off immediately and do not enter the mixing chamber. The impeller retains droplets and stabilizes the aerosol cloud. Any liquid residues left in the system can run off into the siphon through the continuously rising mixing chamber tube. Furthermore, the baffle ball must be permanently attached in a central position relative to the nebulizer. It does not require any readjustment after the mixing chamber nebulizer system is cleaned.



Fig. 14 Nebulizer-mixing-chamber-burner-system

- 1 Burner
- 2 Fixing screw for burner
- 3 Fuel gas supply
- 4 Screw joints of mixing chamber parts5 Locking ring for nebulizer
- 5 Locking ring for nebulizer6 Nebulizer (sample liquid supply)
- 7 Oxidant supply
- 8 Siphon outlet

- 9 Siphon sensor connection
- 10 Siphon
- 11 Siphon sensor
- 12 Mixing chamber head
- 13 Auxiliary oxidant supply
- 14 Safety plug
- 15 Mixing chamber tube



Fig. 15 Mixing chamber and nebulizer, disassembled

- 1 Safety plug
- 2 Mixing chamber tube
- 3 Impeller
- 4 Mixing chamber head with connections for gases, nebulizer and siphon
- 5 Connections for auxiliary oxidant and fuel gas (pointing backwards)
- 6 Nebulizer connection with locking ring
- 7 Baffle ball
- 8 Nebulizer with connection for oxidant and connection for sample tube
- 9 Siphon
- 10 Siphon sensor

#### 3.7.3 Burner and flame type

The novAA 800 can be operated with the following types of flames and their corresponding burners:

- Acetylene-air flame with 50-mm-single-slit burner (universal burner) or 100-mm-single-slit burner for higher sensitivity
- Acetylene-nitrous-oxide flame with a 50-mm-single-slit burner

For laboratory applications that require the determination of elements that are easy to atomize and such that are difficult to atomize, it is advisable to use the 50-mm-single-slit burner (universal burner) because it is not necessary to change this burner between the different measurements.

Uses of the different flame types:

- Acetylene-air flame can be used for most elements
- Acetylene-nitrous-oxide flame is required for difficult-to-atomize elements such as boron, aluminum and silicon.



- 1 50-mm-single-slit burner (universal burner)
- 2 100-mm-single-slit-burner

#### Fig. 16 Burner types

The burners made of titanium are inert with respect to the influences of aggressive sample solutions. The burners can be exchanged easily and can be continuously rotated up to  $90^{\circ}$  in-between 2 stops. One stop is positioned in such a way that the burners are aligned to the optical axis. The  $90^{\circ}$  stop sets the nonsensitive diagonal position of the burners for determining main components.

#### 3.7.4 Sensors

The burner-nebulizer system is checked by various sensors so as to guarantee the operational safety.

- A float switch in the siphon indicates the correct filling level of 80 mm in the water column.
- The burner type is identified by a code that is read by two reflex couplers.
- A UV-sensitive sensor monitors the burning flame.

In addition to the above-mentioned sensors, the mixing chamber is also equipped with a safety plug which will fall out if the flame backfires into the mixing chamber.

The ASpect LS control software evaluates the sensor signals and also monitors the gas pressures and the gas flows as well as the status of the flame.

### 3.8 Accessories for flame technique

#### 3.8.1 Autosamplers AS-F and AS-FD

Manual or automatic sample supply can be employed for the flame technique and for the mercury-hydride technique. Automatic operation and multi-element analysis requires the use of an autosampler. The control software of the novAA 800 sets the parameters and controls the functioning.

The novAA 800 can be operated with the following autosamplers:

- The autosampler AS-F is an automatic autosampler.
- The autosampler AS-FD also has a dilution function.

71	
139 positions	Sample tray with 129 sample positions for 15-mL tubes on the outer track and 10 sample positions for 50-mL tubes on the inner track
54 positions	Sample tray with 54 positions for 50-mL tubes

The autosamplers use sample trays with the same diameter. The following sample tray types are available:

The sample trays should be selected according to the requirements of the analysis:

- Available sample volume
- Type of signal evaluation

The software-controlled autosampler arm reaches all the positions intended for sample-taking. The dipping depth into the sample and the special cups is preset, however, it can be adjusted via the control software.

The novAA 800 supplies the autosamplers with operational voltage. Tray and autosampler arm are driven by stepper motors. The tray is rotated. The autosampler arm can be rotated and lowered by 120 mm.

At the top of the autosampler AS-F next to the sample tray there is a purge cup with an overflow. In the autosampler AS-FD the purge cup is located in a plastic block together with a mixing cup. A diaphragm pump delivers the washing liquid from the supply bottle into the purge cup – this action cleans the dipped cannula by washing it inside and out. During the purging process a second diaphragm pump pumps any excess washing liquid to the waste receptacle located underneath the table.



Fig. 17 Autosampler AS-FD with separate Fluidics module

- 1 Sample tray with cover
- 2 Sampler arm

3

4 Storage bottle for diluent

Storage bottle for washing liquid

- 5 Fluidics module
- Change-over valve with metering syringe (5000  $\mu$ L) 6

The autosampler AS-FD features a separate Fluidics module with a metering syringe (5000  $\mu$ L). The Fluidics module is electrically connected to the autosampler and is supplied with operating voltage via the novAA 800. Standards or samples are diluted in the mixing cup by first placing the concentrate into the mixing cup. Then the diluent is added at a high dosing speed (max. volume: V = 25 mL). A fixed waiting time ensures complete mixing. A diaphragm pump extracts the residual liquid that has not been drawn in by the nebulizer.

The autosampler AS-FD with dilution function provides the following advantages:

- Preparation of standards for calibration by diluting one or several stock standards in the mixing cup
- Dilution of the sample if its concentration is too high, i.e., its element content is higher than 110 % of the calibration standard with the highest concentration
- Dilution of all samples at freely selectable dilution ratios up to a ratio of 1:500

#### 3.8.2 Piston compressor PLANET L-S50-15

If no in-house compressed air supply is available, a compressor should be used to provide the compressed air for the acetylene-air flame.

Analytik Jena offers the piston compressor PLANET L-S50-15 as an optional accessory. The compressed air is free from water, dust and oil. At a maximum operating pressure of 800 kPa and with a 15-L-air cylinder, the compressor is sufficient to meet the requirements for compressed air supply. For installation and maintenance of the device, please observe the information provided in the operating instructions of the piston compressor PLANET L-S50-15.

#### 3.8.3 Injection module SFS 6

The injection module SFS 6 (Segmented Flow Star) is available as an optional accessory. It may be used in combination with an autosampler or in manual mode.

The SFS 6 ensures reproducible conditions in the flame. It permanently draws in purging and carrier solution which allows to keep the burner at a constant temperature. Small sample volumes can be measured in a reproducible manner and gaged against a carrier solution.

The operating principle of the injection module SFS 6 is based on a magnetic valve with two inlets and one outlet to the nebulizer. The sample aspiration tube is located at the energized inlet. It is dipped directly into the sample or is connected to the autosampler cannula. The non-energized inlet is connected to the aspiration tube for the washing or carrier solution.

There are two switching states:

- Basic state: Sample path is blocked, washing solution path is free
- Active state: Sample path is free, washing solution path is blocked

The injection module SFS 6 is controlled by the ASpect LS software.

Connection for the control cable

Short piece of tube to the nebulizer

Tube to the purge solution

1 2

3 4

5

Support

cannula

Sample intake tube



Fig. 18 Injection module SFS 6

#### 3.8.4 Scraper – automatic cleaner of the burner head

It is recommended to use the automatic cleaner of the burner head (scraper) for continuous and fully automated operation with a nitrous oxide flame. When working with the nitrous oxide flame and particularly when working with a flame with very high fuel gas content, as used for determining the elements silicon, tungsten, molybdenum and tin, a carbon deposit will gradually build up on the slit of the burner. If these deposits are not constantly removed, the burner slit will get clogged. This would lead to a low reproducibility of the measurement results.

Once activated in the software and stored as a method parameter, the scraper guarantees a continuous and reproducible measuring process without any disturbances and interruptions. The automatic cleaning of the burner head varies with the composition of the flame and the analysis task. On the other hand, the scraper can also be used for the automation of the burn-in process of the nitrous oxide flame. When activated in the FLAME / CONTROL window, a cleaning step is executed every 30 s.

The scraper is fixed to the burner head with two knurled head screws. It can be detached if it is not needed. The scraper can be retrofitted to a 50-mm burner.

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Fig. 19 Scraper mounted to a 50-mm burner head

# 3.9 Optional accessory – Air Purge Kit

The Air Purge Kit (APK) is used in combination with the atomic absorption spectrometers of the novAA series (novAA 800, novAA 400 P Flame) to purge the spectrometer. The purging process with purified and dried air prevents the ingress of dust and corroding vapors into the optical area of the spectrometer. This significantly improves the quality of the chemical analysis and the service life of the spectrometer even under difficult ambient conditions.

For a description of the Air Purge Kit, please refer to the corresponding manual of the accessory.

# 3.10 Optional accessory – hydride systems

The hydride systems available range from simple batch systems for users with small sample volumes through to fully automated continuous devices with flow injection.

HS 50:	Most simple batch system with pneumatic operating principle. The quartz cell is heated by the acetylene-air flame.
HS 55 modular:	Batch system with electrically heated cell unit with or without "Hg Plus" module for mercury detection. The reduction agent solution is metered by a 1-channel hose pump.
HS 60 modular:	Hydride system for continuous flow injection operation with electrically heated cell unit with or without "Hg Plus" module for mercury detection.

For a more detailed description of the hydride systems, please refer to the respective accessory manuals.

- 1 Connection cable for scraper
- 2 Knurled head screw
- 3 Scraper
- 4 Fixing screw for burner
- 5 Knurled head screw
- 6 50-mm burner head

# 4 Installation and initial start-up



# CAUTION

Prevent any unauthorized interference!

The device may only be assembled, installed and repaired by service engineers from Analytik Jena or by technical personnel authorized by Analytik Jena.



# CAUTION

Observe the safety instructions!

When installing and starting up the device, please observe the instructions provided in the section "Safety instructions" on page 9. Compliance with these safety instructions is a requirement for the error-free installation and the proper functioning of your AAS measuring environment. Always observe all warnings and instructions which are displayed on the device itself or which are displayed by the control and analysis software ASpect LS.

The novAA 800 will be delivered directly to the final instrument location by a transport company. The delivery by this company requires the presence of a person responsible for the instrument installation.

All persons designated for operating the device are obliged to attend the briefing of the Analytik Jena service department.

The customer must ensure that the installation conditions at the place of installation comply with the specifications stipulated by Analytik Jena ( $\rightarrow$  section "Installation conditions" on page 38).

# 4.1 Installation conditions



## NOTICE

The device may only be assembled, installed and repaired by service engineers from Analytik Jena or by technical personnel authorized by Analytik Jena. Any unauthorized interference limits warranty entitlements.

The assistance of one person is required during some stages of the installation process. The service engineer will test the device and document the test in the test report of the novAA 800.

The operator must provide everything that is not included in the original delivery, but required for operating the novAA 800. Operation of the novAA 800 demands certain local and system-specific requirements:

- Suitable place for assembly
- Space requirement
- Environmental conditions
- Supply of inert gas, fuel gas and oxidant

- Exhaust unit
- Mains connection



## CAUTION

Pay attention to the safety instructions in the chapter "Safety instructions" on page 9. Observe work protection regulations. Warnings regarding potential dangers do not replace valid work protection regulations!

Possible dangers when working with the novAA 800 are:

- Risk of burning by flame and hot burner parts
- Danger from electric current
- Danger of UV radiation
- Danger of ozone or nitride oxide formation
- Danger when handling compressed gas cylinders
- Danger from toxic and chemically aggressive substances

#### 4.1.1 Environmental conditions

The novAA 800 may only be operated within closed rooms. The location must be set up like a chemical laboratory. The location must meet the following conditions:

- The installation site must be devoid of dust, draft air currents, vibrations and caustic fumes.
- Do not place the novAA 800 near sources of electromagnetic interference.
- Avoid direct sunlight and heater radiation on the novAA 800. In extreme cases, provide air conditioning in the room.
- A separate room is recommended for sample preparation and storing chemicals.

The following ambient conditions must be met by the operating room of the no-vAA 800:

Temperature range	+5 °C to +40 °C
Humidity during operation	Max. 90 % at 40 °C
Transport temperature (desiccant)	-40 ℃ to +70 ℃
Air pressure	0.7 bar to 1.06 bar
Recommended max. altitude	2000 m

The ambient conditions for the novAA 800 are identical for operation and storage.

## 4.1.2 Energy supply



## WARNING

Observe the mains connection!

During electrical installation, observe any local regulations! The mains supply must be correctly earthed. Do not use an adapter in the mains cabling.

novAA 800 D + G The models novAA 800 D and novAA 800 G are operated on a single-phase alternating current network. The current load at the maximum heating rate can reach up to 40 A for a short period (1 s). The mains voltage at the novAA 800 P should not decrease by more than 6 % during this period. In case of deviations from these values, please contact the Analytik Jena service department. Appropriate accessories can be supplied.

> Optimum device function strongly depends on a correct mains connection with adequate cable cross-section. The mains connection must be protected by a 35 A slowblow fuse on the input (building) side and must be installed prior to delivery of the novAA 800 near the assembly point. The length of the instrument cable is 3 m. The CEE surface socket (two-pole + E Blue 5UR 3 206-2 220/32, Siemens) is supplied according to the terms of delivery.

The auxiliary components that are to communicate with each other, for example PC, monitor, printer and hydride system, have to be connected to the power strip that is supplied with the device. The power strip is plugged into the rear of the novAA 800 D and G and is connected to the same phase as the base device itself. The compressor needs a separate power supply.

If you use your own PC printer configuration, and if it is connected via the 5-socket power strip, please observe the limit of the permitted line current. To avoid sudden voltage fluctuations, do not connect the novAA 800 to the same electrical circuit as other power-intensive devices.

Supply voltage Frequency	230 V ~ 50 / 60 Hz							
Mains fuse installation in the building	35 A, safety fuse, slow blow, single phased							
Power consumption	2600 VA (basic device: 1400 VA + output socket: 1200 VA)							
Maximum current consumption	28 A for a period of 8 s or 40 A for 1 s							
Output socket	Same as input voltage							
	For connection of accessories: PC, monitor, printer, hydride system, cooling unit							
Power consumption of the hydride system	650 VA while heating the cell 400 VA in continuous operation							

novAA 800 F

Conditions for switching on

The novAA 800 F is operated on single-phase alternating current. Optimum device function strongly depends on a correct mains connection. The mains connection must be protected by a 16 A slow-blow fuse on the input (building) side. The length of the instrument cable is 2 m.

Conditions for switching on

The auxiliary components that are to communicate with each other, for example PC, monitor, printer and hydride system, have to be connected to the power strip that is supplied with the device. The power strip is plugged into the rear of the novAA 800 F and is connected to the same phase as the base device itself. The compressor and cooling unit need a separate power supply.

If you use your own PC printer configuration and connect the printer to the 5-socket power strip, observe the maximum permissible operating current. To avoid sudden voltage fluctuations, do not connect the novAA 800 to the same electrical circuit as other power-intensive devices.

Supply voltage Frequency	230 V ~ 50 / 60 Hz
Mains fuse installation in the building	16 A, single phase
Power consumption	1350 VA (basic device: 150 VA + output socket: 1200 VA)
Output socket	Same as input voltage For connection of accessories: PC, monitor, printer, hydride system
Power consumption of the hydride system	650 VA while heating the cell 400 VA in continuous operation

## 4.1.3 Gas supply



#### WARNING

Risk of explosion due to leaking acetylene! Risk of the build-up of an oxygen-deficient atmosphere caused by leaking gas!

The operator must ensure that the connector type used on the outlet side of the gas pressure controller complies with the national requirements.

The operator must carry out the necessary safety leakage tests on all gas supply lines and connectors including those on the device on a weekly basis. For this, possible pressure losses from closed systems and lines under pressure are to be determined. The leak is to be localized and corrected immediately. If the gas is supplied via compressed gas cylinders, these must be attached to the wall with cylinder mounts in an upright position outside the laboratory.

Gases in the graphite tube technique

The inert gas argon is used to protect the graphite components of the atomizer which are subjected to extreme temperatures. The inert gas is also used as a means of transport for the pyrolysis components accrued during the analysis. The purity of the inert gas is extremely important for the analysis and for the useful life of the graphite tube.

Introducing an auxiliary gas during the pyrolysis (e.g. compressed air), can accelerate the ashing of the sample, i.e. the removal of the matrix components. The auxiliary gas is fed in through the "Gas Auxiliary" connection (item 2 in Fig. 28 on page 50) at the rear of the device.

The inlet pressure on the spectrometer must be 600 to 700 kPa.

Gases in

flame technique

The pressure hose for argon is included in the delivery. The standard length of the hose is 5 m. Other hose lengths can be provided upon request. Please contact the Analytik Jena service department.

Recommended	inert gas	Inlet pressure	Consumption				
Argon 4.8 or sup Permitted comp	perior oonents:	600 to 700 kPa	Max. 2 L/min (depending on the tempera-				
Oxygen≤3 ppm	I		ture-time program)				
nitrogen	$\leq$ 10 ppm						
hydrocarbons	≤ 0.5 ppm						
humidity	≤ 5 ppm						
Auxiliary gas: Co free, particle-fre	ompressed air, grease- ee	600 to 700 kPa					

The flame technique requires an oxidant (compressed air or nitrous oxide) and acetylene as a fuel gas. On request Analytik Jena can also supply accessories to use propane as a fuel gas.

The purity of the gases is extremely important for the analysis. The piston compressor PLANET L-S50-15 can be used to supply the compressed air. If compressed air is supplied via an in-house compressed air supply system, please consult the service department at Analytik Jena. Nitrous oxide and acetylene are supplied via compressed gas cylinders or an in-house supply system.

The pressure hoses are supplied. The pressure reducing valves are optional.

- Length of the hose for cylinder connection 5 m
- Length of the hose for compressor
   5 m

It is also possible to connect other hose lengths. Please consult the service department at Analytik Jena.

Fuel gas and oxidant	Inlet pressure	Consumption
Compressed air, oil-free, grease-free, particle-free	400 to 600 kPa	Max. 825 NL/h
$N_2O$ , oil-free, grease-free, purity 2.5	400 to 600 kPa	Max. 660 NL/h
Acetylene Purity 2.6 (for flame photometry): Superior to 99.5 Vol% relative to $C_2H_2$ , without acetone	80 to 160 kPa	Max. 315 NL/h

## 4.1.4 Exhaust unit



#### CAUTION

Risk of poisoning due to leaking gases!

Switch on the exhaust unit prior to starting the novAA 800. Extract the exhaust air from the laboratory and prevent congestion!

Correct extraction can only be ensured by an exhaust hood that is installed directly above the sample chamber.

The exhaust unit should remove health-damaging combustion residues of the flame as well as any ozone that is produced during combustion. Ozone is produced when air reacts with UV radiation from the hollow cathode lamps and the burner flame. Use a suction device made of heat-resistant and corrosion-resistant material. The first 6 m of the exhaust air system should be made of metal.

Parameter	Properties
Material	V2A
Exhaust performance for graphite tube technique	Approx. 1 m <sup>3</sup> /min
Exhaust performance for nitrous oxide flame	Approx. 8 to 10 m <sup>3</sup> /min
Exhaust performance for air flame	Approx. 5 m <sup>3</sup> /min
Hood opening	Approx. 300 x 300 mm
Distance to the upper edge of the device	Approx. 200 to 300 mm
Tube diameter	Approx. 100 to 120 mm

#### 4.1.5 Space requirement, weight and device layout

The novAA 800 is a compact device conceived to be mounted on a table. The required space depends on the number of components needed for measurement. When placing the device and system components observe a minimum clearance of 15 cm to walls and neighboring installations.

The PC with the monitor, the printer and the keyboard are arranged beside the base device. The PC and printer may also be placed on a separate side table.

The workbench must be positioned in a way that allows easy access from all sides. In addition to that, the workbench must meet the following requirements:

- Minimum dimensions: 1800 x 700 mm, select the height based on ergonomic aspects.
- Load capacity of the workbench: min. 180 kg
- Table surfaces: resistant to wiping, scraping, corrosion and water

The autosamplers for flame mode AS-F or AS-FD are mounted in the sample chamber of the novAA 800. The storage bottle for the washing liquid of the AS-F or the Fluidics module of the AS-FD are placed next to the AAS device.

The autosampler AS-GF for the graphite furnace technique is mounted in the sample chamber.

The accessories for the hydride technique (e.g. HS 60 modular) are placed on a separate table in front of the novAA 800. The Air Purge Kit can be placed next to the novAA 800 or on a separate side table (length of the connecting hose: 2 m).

The following are located on the floor near the device:

Cooling unit KM 5

The mobile cooling unit KM 5 must be placed with a minimum clearance of 15 cm to both sides to ensure optimum circulation of the cooling air inflow and outflow.

- The collecting bottle for residual sample liquid, residual autosampler washing liquid and residual liquid of the hydride system
- The piston compressor PLANET L-S50-15 (only for flame technique)

Components	Width [mm]	Height [mm]	Depth [mm]	Weight [kg]
On the workbench				
novAA 800	820	600	770	D: 130 G: 125 F: 95
AS-GF	250	550	380	7.2
AS-F	340	350	460	6.5
AS-FD				
Sampler	340	350	460	6.5
Fluidics module	360	310	165	3.5
HS 60 modular	360	370	240	14
HS 55 modular	360	370	240	14
HS 50	270	210	190	2
Air Purge Kit (APK)	245	265	260	3.2
Under the workbench				
Cooling unit KM 5	300	600	500	32
Compressor PLANET L-S50-15	Ø 400	490		27
Waste bottle	Ø 200	400		



Fig. 20 Dimensions of the novAA 800 - front view



Fig. 21 Dimensions of the novAA 800 (with AS-FD and Fluidics module)



Fig. 22 Dimensions of the novAA 800 (with autosampler AS-GF)



Fig. 23 Installation diagram novAA 800 with exhaust unit

# 4.2 Supply and control connections

The supply lines are connected by the technicians of the Analytik Jena service department during the installation of the novAA 800.

The mains switch is located on the right side of the novAA 800. The right device side also contains an easily accessible connection panel with interfaces for PC and accessories. The media connections for gases, cooling water and electricity as well as the fuses are located on the rear panel. The mains connection for the power strip supplied to connect the accessories is equally located at the rear.

A pair of carrying handles is fastened to the left and the right for transport and installation. After installation the handles are unscrewed and the openings sealed with the plugs supplied with the device.



Fig. 24 novAA 800 - side view with carrying handles

- 1 Clamp for fastening the device cover
- 2 Connections for PC and accessories (see below)
- 3 Carrying handles
- 4 Mains switch



Fig. 25 Supply and control connection panel

<ul> <li></li> <li><!--</td--><td>* * * *</td><td></td></li></ul>	* * * *	

Fig. 26 Rear view novAA 800 with connections and fuses

- 1 Connection compressed air
- 2 Connection fuel gas (C<sub>2</sub>H<sub>2</sub>)
- 3 Connection nitrous oxide (N<sub>2</sub>O)
- 4 Type plate

- 5 Gas and cooling water connection (see Fig. 28)
- 6 Fuses and electrical connections (see Fig. 27)





Fig. 27 Fuses and electrical connections

#### novAA 800 D + G:

- 1 Fuses F3-F9
- 2 Mains connection for accessories (with provided 2 power strip) 3
- 3 Mains connection line for novAA 800
- 4 Fuses F1, F2

#### novAA 800 F:

- 1 Fuses F3-F7
- 2 Fuses F1, F2
- 3 Mains connection for novAA 800
- 4 Mains connection for accessories (with provided power strip)

The novAA 800 D is equipped with connections for the following gases: Inert gas (argon) and auxiliary gas (e.g. compressed air) for graphite furnace technique and fuel gas (acetylene), nitrous oxide and compressed air for flame technique. The novAA 800 G does not have any connections for the flame gases. The novAA 800 F does not have the connections for inert gas and auxiliary gas.



Fig. 28 Gas and cooling water connections

- 1 Connection inert gas (argon)
- 2 Connection auxiliary gas
- 3 Connection Air Purge Kit
- 4 Cooling water return flow "Water out"
- 5 Cooling water inlet "Water in"

Type plate

The type plate is located on the rear of the device. The type plate contains the serial number and the electrical connection data.

Information on the type plate	novAA 800 D + G	novAA 800 F						
Manufacturer (with address)	Analytik Jena, 07745 Jena, Konrad Zuse Str. 1, Germany							
CE marking								
Symbol for waste disposal in accordance with the WEEE directive (2002/96/EC)	Meaning: Do not dispose in domestic waste!							
Device type and model	AAS novAA 800 D AAS novAA 800 G	AAS novAA 800 F						
Voltage / Frequency	230 V ~ 50 / 60 Hz	230 V ~ 50 / 60 Hz						
Power consumption	2600 VA	1350 VA						
Max. current consumption	max. 28 A/8 s or max. 40 A/1 s	n/a						
Serial number	S-NR 10-1430D-AQ	XXX (D – model specification)						

The serial number is also attached to the top of the lamp chamber.

# 4.3 Removing the transport locks



#### NOTICE

Removing the transport locks! The transport locks must be removed by Analytik Jena service engineers or technical personnel authorized by Analytik Jena during the initial device installation.

Transport lock on the monochromator

For transportation, the monochromator of the novAA 800 is protected by a securing device. In addition to that, the graphite furnace of the combined device novAA 800 D and the novAA 800 G is tilted backwards and secured in parking position by a transport lock (red foam).

The two transport locks must be removed prior to commissioning the novAA 800.



Fig. 29 Transport lock on the novAA 800

- 1. Unscrew and remove the clamps of the device cover on the left and right side walls (item 1 in Fig. 24).
- 2. Remove the device cover.
- 3. Unscrew the red-marked transport lock (arrow in Fig. 29) from the mesh lever and remove it from the spectrometer chamber.

Retain the transport locks for future use.

- 4. Cover the opening in the photometer hood with non-transparent black adhesive tape.
- 5. Place the device cover and fasten the clamps on the left and right side walls.



Fig. 30 Transport locks on the graphite furnace

 Pull out the red and white transport lock from the sample chamber of the novAA 800 D or the novAA 800 G and retain them for future use.

After unlocking the graphite furnace it can be tilted forwards into the optical axis ( $\rightarrow$  section "Changing the atomization technique" on page 60).

Transport locks on the graphite furnace

# 4.4 Placing and connecting the novAA 800

#### Tools

- 4 plugs, plastic (supplied with the device)
- 17-mm open-end wrench
- 1. Unscrew and remove the four handles and retain them for future use.
- 2. Seal the openings with plugs.
- Install the gas supply on the rear side of the device (→ section "Installing the cooling unit")

The mobile cooling unit KM 5 is required to use the graphite furnace technique on the novAA 800 D and the novAA 800 G. For detailed information on installation, operation and maintenance, refer to the operating instructions "Mobile Cooling Unit KM 5" supplied with the device.

- 1. Fill the mobile cooling unit KM 5 ( $\rightarrow$  section "Cooling unit KM 5" on page 112).
- Set up the cooling circuit: Push the tube connector onto the novAA 800 and the KM 5.

On the KM 5 (bottom): "Water inlet" ► On the novAA 800: "IN"

On the KM 5 (top): "Water return flow" ► On the novAA 800: "OUT"

3. Connect the control cable of the KM 5 to the correspondingly marked connector on the right wall of the novAA 800 (item 5 in Fig. 25 on page 49).

**Note:** The service button of the KM 5 remains in "OFF" position, i.e. the green operating light is not illuminated. This is the only way to ensure that the mobile cooling unit can be controlled by the novAA 800 control software.

4. Bleed the cooling circuit ( $\rightarrow$  Section "Cooling unit KM 5" on page 112).

# 4.5 Installing and starting the ASpect LS software

Installing and launching the software program ASpect LS are described in the "ASpect LS" manual.

# 4.6 Loading the lamp turret and adjusting the lamps



#### WARNING

Risk of skin and eye damage caused by UV radiation! Turn off the lamp current before opening the lamp chamber.

To do so, open the section OPTICAL PARAMETERS in the SPECTROMETER / CONTROL window of the ASpect LS software and set the lamp current [mA] to zero. Open the drop-down list BACKGROUND CORRECTION and select the option NO BACKGROUND. Click [SET]. Negate the error message.



# CAUTION

Risk of burns! Allow lamps to cool down before replacing them.

# NOTICE

Risk of damage to the lamp! Do not touch the lamp window. Remove and install lamps only when the device is de-energized.

The 8-lamp turret should preferably be loaded with coded hollow cathode lamps. The use of uncoded lamps is possible.

The lamp turret is usually de-energized before it is loaded. However, lamps can also be loaded or replaced while the turret is energized.



- 1 Bracket of the deuterium hollow cathode lamp
- 2 Deuterium hollow cathode lamp
- 3 Base plate of the lamp turret
- 4 Hollow cathode lamp (position of the lamp turret for removal and installation)
- 5 Tension spring

Fig. 31 Arrangement of the lamp turret and bracket of the D<sub>2</sub>-HCL

The 8-lamp turret and the deuterium hollow cathode lamp are located behind the left front door in the lamp chamber of the novAA 800. The unit can also be accessed via the left side wall after removing the cover plate.



Fig. 32 Cover plate on the left side panel

## 4.6.1 Removing and installing the hollow cathode lamp

In de-energized state	1. Open the door to the lamp chamber.
	2. Turn the lamp turret until you can access the position you want to load.
	3. Hold the lamp turret and disengage the tension spring.
	4. Remove the lamp from the lamp socket. Do not touch the lamp window!
	5. Plug the new lamp into the lamp socket.
	6. Hold the lamp turret and attach the tension spring.
In energized state	In energized state, it is possible to move the lamp turret to the desired position using the control software. Furthermore, it is possible to configure uncoded lamps for mounting and dismounting in ASpect LS ( $\rightarrow$ section "Configuring the lamp turret in ASpect LS" on page 56).
	1. Click the 🖆 icon to open the SPECTROMETER window and go to the CONTROL tab.
	2. Use the [LAMP TURRET] button to open the corresponding window.
	3. Highlight the position that is to be mounted with a lamp or which is to be changed
	4. Click [CHANGE LAMP] to move the lamp turret to the desired position.
	5. Replace the lamp (see above).
	6. Configure the uncoded lamp in ASpect LS.
	7. Exit the LAMP TURRET window by clicking on [CLOSE].
	$\checkmark$ The lamp turret is automatically initialized with the newly loaded lamps.
4.6.2 Removing a	and installing the deuterium hollow cathode lamp
	1. Open the lamp chamber door.

- 2. Unscrew the fastening nuts (items 1, 3, 5 in Fig. 33) and remove the lamp holder from the lamp chamber.
- 3. Loosen and remove the safety bracket (item 5 in Fig. 34). Pull the lamp socket from the lamp.
- 4. Carefully pull out the lamp from under the tension spring (item 1 in Fig. 34).
- 5. Carefully place the new lamp under the tension spring and push it to the stop (item 2 in Fig. 34).

Note: Do not touch the lamp window!

- 6. Attach the lamp socket to the lamp. Push the safety bracket until it reaches the lamp socket and screw it to the holder
- 7. Adjust the lamp axis parallel to the mount of the holder (by eye): Use the two adjustment screws to change the position of the lamp (item 4 in Fig. 34).
- 8. Insert the holder to the lamp chamber and lightly screw in the nuts.

Note: The nuts are only tightened by hand after the adjustment.

1,3,5

4

Securing nuts of the lamp holder 2 Adjustment screws

> safety bracket of the lamp holder

Attachment screws for the



Fig. 33 D<sub>2</sub>-HCL holder installed in the lamp chamber



 $Fig. \ 34 \qquad D_2\text{-HCL with holder, removed from the lamp chamber}$ 

1 Tension spring

- 2 Stop
- 3 Support

4 Adjustment screw

5 Safety bracket of the lamp socket

4.6.3 Configuring the lamp turret in ASpect LS

Coded lamps

If coded lamps are available, the relevant data for the method of analysis which are saved on the transponder, such as the lamp type, elements, maximum and recommended lamp current as well as maximum and recommended boost current, are read during initialization and assigned to the lamp position. Uncoded lamps



#### NOTICE

Observe the lamp position! If you use uncoded hollow cathode lamps, make sure that the position data in the software matches the actual loading position in the turret.

- 1. Click the 🚈 icon to open the SPECTROMETER window and go to the CONTROL tab.
- 2. Use the [LAMP TURRET] button to open the corresponding window.
- 3. Highlight the position that is to be mounted with a lamp or which is to be changed.
- 4. Use [CHANGE] to open the SELECT LAMP/ELEMENT window.

Select	lam	p/el	eme	nt																		-		×
Lamp	pos	sitio	n:		1			amp MHC	type CL	,	~	Ma	ax. la	amp	curre	ent (m	nA]:	10	.0	€ N	lax. Boost [m	nA]:	0.0	
Eleme	ents	-																		Elem.		Name		
L	i E	3e											В	С	Ν	0	F	Ne		Cr Mn	Chromium (	Cr) e (Mn)		
N	a N	<b>l</b> g											Al	Si	Ρ	S	CI	Ar		Fe	Iron (Fe)			
K		Ca	Sc	Ti	۷	Cr	Mn	Fe	Со	Ni	Cu	Zn	Ga	Ge	As	Se	Br	Kr		Ni	Nickel (Ni)			
R	b	Sr	Y	Zr	Nb	Мо	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Te	1	Xe		Cu	Copper (Cu	I)		
C	s E	3a	La	Hf	Та	W	Re	0s	lr.	Pt	Au	Hg	TI	Pb	Bi	Po	At	Rn						
F	r F	Ra	Ac	_		_	_				-			1										
		-	Ce	Pr	Nd	Pm	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu								
			Ιh	Pa	0	Np	Pu	Am	Cm	Bk	Ct	Es	⊦m	Md	No	Lr								
I.																		^						
																		×						
																					ОК		Cance	I

#### Fig. 35 Window SELECT LAMP/ELEMENT

#### 5. Enter the following values:

LAMP POSITION	Shows the position in the lamp turret. Cannot be edited in this window.				
LAMP ТҮРЕ	For selecting the lamp type. NONE Position does not contain a lamp				
	HCL Single-element hollow cathode lamp				
	MHCL Multi-element hollow cathode lamp				
MAX. CURRENTS	For setting the maximum lamp current.				
Elements	Click the element symbol in the periodic table to select the lamp ele- ment:				
	<ul> <li>Blue buttons indicate selectable elements.</li> </ul>				
	<ul> <li>Gray (inactive) buttons indicate elements that cannot be analyzed using the AAS technique.</li> </ul>				
	<ul> <li>Green buttons indicate selected elements.</li> </ul>				

For M-HCL it is possible to select several elements. Clicking the symbol of the element again will clear the selection. Selected elements are shown in the adjacent table.

6. Click [OK] to exit the SELECT LAMP/ELEMENT window and return to the LAMP TURRET window.

The new lamp specification is automatically loaded to the table of the LAMP TURRET window.

- 7. Exit the LAMP TURRET window by clicking [CLOSE].
  - ✓ The lamp turret is automatically initialized with the newly loaded lamps.

#### 4.6.4 Adjusting the lamps

Fine adjusting the lamps is generally required only once after a new installation of the lamp.

The useful life of the lamp strongly depends on the current setting for the lamp. The recommended operating current varies from lamp type to lamp type. For the following adjustment, observe the instructions in the cookbook of the ASpect LS software, the Analytik Jena operating instructions for the various lamps and the information supplied with the lamp.

Adjusting the line source

Maximizing the

useful life of the lamp

- 1. Click the  $\Delta$  icon to open the SPECTROMETER window and go to the CONTROL tab.
- Use the [LAMP TURRET] button to open the corresponding window.
  - (D) Lamp turret П  $\times$ Mounting Lamp history Code lamps Max. curr. Max Boost Recmd, curr, Recmd, boos Alid Pos Туре cod. Flements [mA] [mA] [mA] [mA] adj. MHCL -Cr;Mn;Fe;Co;Ni;Cu 10.0 1 2 MHCL Cd;Pb 10.0 + \* 3 HCL Dy 10.0 \* Na;K;Cr;Hg 4 MHCL 10.0 10.0 HCI 6 MHCL Ca;Tl;Pb;Bi 10.0 \* 7 MHCL Rh 10.0 \* -10.0 8 HCL -< Change Register lamp Unregister lamp Initialize Delete table Lamp alignment Energy Align 0 change lamp Close
  - Fig. 36 Window LAMP TURRET
  - 3. Highlight the lamp to be adjusted in the table.
  - 4. Click the [ALIGN] button.

The lamp is then automatically adjusted relative to a pitch circle. In the lamp alignment area, the energy is displayed as a **blue** bar during the adjustment.

Adjusting the deuterium hollow cathode lamp

- 1. Click the 🚈 icon to open the SPECTROMETER window and go to the CONTROL tab.
- 2. Click the [ELEMENT] button to open the SELECT ELEMENT/LINE window.
- 3. Click on the blue button to select the element. Select the wavelength range 190-350 nm from the Line table. Click on [OK] to close the window.
- 4. Open the drop-down list BACKGROUND CORRECTION and select the option ONLY D2 BACKGROUND.
- 5. Approach the spectrometer parameters using [SET].
- 6. Open the ENERGY tab.

<b>∕</b> E Spectrometer		_		×
Control Energy Energy scan Spectrum				
Energy levels	Parameters AGC Peak pick Transfer to method			
D2HCL: 125098	D2HCL current [mA]: 0.7			
Start	Lamp alignment Align 0 Energy			
		Clo	se	

Fig. 37 Window SPECTROMETER – ENERGY

- 7. Initiate the energy measurement by clicking on [START]. Wait until the D2-HCL has started.
- 8. Set the energy level to a maximum value:
- With focus adjusting: Move the lamp holder up or down slightly before tightening the securing nuts (items 1, 3, 5 in Fig. 33 on page 56).
- With axis adjustment: Adjust the adjustment screws (item 2 in Fig. 33).
- 9. Stop the adjustment process by clicking on [STOP] and click [CLOSE] to exit the dialog.
  - ✓ The  $D_2$ -HCL is adjusted.

#### 4.7 Changing the atomization technique



## NOTICE

Always switch off the novAA 800 before installing or removing the autosampler or the hydride system! Connecting or disconnecting electrical contacts may damage the sensitive electronic components of the novAA 800.

novAA 800 D

In the combined device novAA 800 D the flame atomizer and the graphite furnace are both attached to a bracket on the height adjustment that can be tilted by 60°.

Proceed as follows to change the atomization technique:

- Push up the locking lever (item 1 in Fig. 38) and unlatch it.
- When doing so, use the tilt lever (item 2 in Fig. 38) to tilt the graphite furnace backwards or forwards until it reaches the stop. Bring the atomizer into the desired working position.

Locking lever Tilt lever



Changing the atomization technique Fig. 38

For the flame technique, it is possible to adjust the position of the stop using the setscrew ( $\rightarrow$  section "Aligning the atomizer within the beam path" on page 103).

Changing from one atomization technique to the next only requires a few additional steps such as removing the autosampler.

- 1. Close the control software ASpect LS. Shut down the PC and novAA 800. Uninstall the autosampler AS-GF and remove the depth-adjustable stop.
- 2. Tilt the graphite furnace backwards to bring the mixing-chamber-nebulizer system into working position.
- 3. Fill the siphon with water via the mixing chamber tube until the water starts running off via the outlet tube.
- 4. Install the burner on the mixing chamber tube and clamp it to the stop.
- 5. Mount the safety glass pane.

Changing from graphite furnace to flame atomizer Changing from flame atomizer to graphite

furnace

- 6. Install the autosampler AS-F / AS-FD if required.
- 7. First switch on the PC, then the novAA 800. Start ASpect LS.
- 8. Open the window QUICKSTART to select flame technique. Initialize the device.
- 1. Take off the safety glass plane.
- 2. Remove the burner.

**Caution!** Risk of burns from touching the hot burner! Observe the required cooling times.



## NOTICE

After removal of the burner, close the mixing chamber tube with the safety stopper. Otherwise, acid fumes could escape from the siphon and damage the novAA 800. Furthermore, the water in the siphon can not leak out when you tilt the mixing-chambernebulizer system backwards.

- 3. Tilt the mixing-chamber-nebulizer system backwards to bring the graphite furnace into operating position.
- 4. Attach the stop for the autosampler AS-GF.
- 5. Shut down ASpect LS. Switch off the PC and then the novAA 800.
- 6. Install the AS-GF.
- 7. First switch on the PC, then the novAA 800. Start ASpect LS.
- 8. Open the window QUICKSTART to select the graphite furnace technique. Initialize the device.
- 9. Adjust the AS-GF.

The individual steps for the installation and adjustment of the autosampler AS-GF are described below in more detail.

#### novAA 800 G The novAA 800 G can be used for graphite furnace, HydrEA and hydride technique. Together with a clamped connector, the graphite furnace is attached to a height adjustable bracket that can be tilted by 60°. The connecting piece serves as a receptacle for the cell unit of the hydride system.

To change over to the hydride technique:

- Push up the locking lever (item 1 in Fig. 38) and unlatch it.
- When doing so, use the tilt lever (item 2 in Fig. 38) to tilt the graphite furnace backwards until it reaches the stop. Bring the clamped connector into the desired operating position.
- Attach the cell unit to the connector and use the attachment screws to lock it in that position.
- Follow the separate operating instructions to install the hydride system. Take care to switch off the novAA 800 before installing the hydride system.
- First switch on the PC, then the novAA 800.
- Open the window QUICKSTART in the ASpect LS to select the hydride technique. Initialize the device.

# 4.8 Graphite tube technique

# 

# 4.8.1 Connections in the sample chamber for the graphite tube technique

Fig. 39 Elements in the sample chamber for the graphite furnace technique

- 1 Plastic pin for attaching the safety glass pane (flame technique only)
- 3 Graphite furnace with connections
- 4 Height-adjustable stop for AS-GF
- 5 AS-GF hinge on the right sample chamber
- 2 AS-GF receptacle on the right sample chamber



Fig. 40 Connections on the graphite furnace

- 1 Cooling water connections: red hoses
- 2 Furnace window
- 3 Radiation sensor
- 4 Gas connections:
- white and black hoses

- 5 Sensor connection for cooling water temperature
- 6 Power cable
- 7 Fuse on the graphite furnace
- 8 Dosing opening with graphite funnel
- 9 Furnace clamps with electrodes

The connections for gas, cooling water and electric current are permanently installed on the graphite tube furnace.

# 4.8.2 Default settings in the software for the graphite technique

Open the QUICKSTART window in the ASpect LS software to set the options for the graphite furnace technique. The user interface of the software is updated with the method and device parameters after initialization.

PERATOR:	SuStein						
.AB.:	TecDoc						
ECHNIQUE:	Graphite furnace	(Platform)	~				
Worksheet		Last changed	Ву	Technique	^	DESCRIPTION	
Ag in strong matri	x	03.04.2020 15:14	Analytik Jena	Graphite furnace (Wall)		Sample preparation: Elem./Wavelength: Ag 328.07 nm	
As in aqueous solution (Hy)		03.04.2020 15:00	Analytik Jena	Hydride		Measurement details: Modifier: Pd/Mg(NO3)2	
As in aqueous solution (HyEA)		03.04.2020 15:02	Analytik Jena	HydrEA		Sample volume: 20 µL Temperature: 650/1700 °C	
Ba in strong matrix		03.04.2020 15:15	Analytik Jena	Graphite furnace (Wall)		Calibration range: 0 - 2.5 µg/L	
Cu in aqueous so	lution	03.04.2020 14:56	Analytik Jena	Flame			
Cu in strong matri	ĸ	03.04.2020 15:15	Analytik Jena	Graphite furnace (Wall)		-	
Hg in aqueous solution (Hy)		03.04.2020 14:59	Analytik Jena	Hydride		Select matching tube type	
Hg in aqueous so	olution (HyEA)	03.04.2020 15:02	Analytik Jena	HydrEA	~		
Favorites Rece	nt Predefined All		,C al	I (12)			
						System check	

Fig. 41 QUICKSTART window in ASpect LS

The height of the graphite furnace is automatically adjusted to the different graphite furnace models (automatic detection).

## 4.8.3 Inserting the graphite tube into the furnace



## NOTICE

The graphite tubes of the novAA 800 are custom-made and must be ordered from Analytik Jena. Do not use any other graphite tube. Other graphite tubes can damage the novAA 800.

Never touch the graphite tube with your bare fingers! Fingerprints burn into the surface, and this causes premature damage to the pyrolysis coating of the tube.

Inserting the graphite tube

1. Use the  $\bigcirc$  button in the ASpect LS to open the FURNACE window. Go to the CONTROL tab.

G Furnace			-		×
Furnace program Modif.Extra	s Optimization Plot Co	ntrol Position			
Graphite furnace Type: Heat cycles: Life time: R	Platform 0 0 eset mation	Furnace Open furnace Cooling water temp. ['C Furnace LED	]: 30		
Clean furnace Temp. ['C]: Ramp ['C/s]: Hold [s]: Water cooler auto	2450 500 5 Start permanent	Temperature for LED sw Test Water flow Inert gas Line frequency Gas box	itch-off:	Deratur Ip.	
Line:	~		OK Ca	ncel	

Fig. 42 Dialog window Furnace / Control

- 2. Use the [OPEN FURNACE] button to open the graphite furnace.
- 3. Use tweezers to insert the graphite tube into the graphite tube furnace so that it is loosely seated on the supports of the furnace shroud and the pipetting opening faces upwards. Wear gloves when inserting the tube by hand.
- 4. Use the [CLOSE FURNACE] button to close the graphite furnace.
- 5. Enter the parameters HEAT CYCLES and LIFE TIME for the inserted graphite tube in the section GRAPHITE FURNACE.
  - ✓ The graphite tube is inserted into the furnace.



Fig. 43 Opened graphite furnace with graphite tube

- 1 Furnace window
- 2 Stationary part of the furnace
- 3 Graphite tube, inserted
- 4 Dosing opening with graphite funnel
- 5 Furnace shroud

- 6 Furnace window
- 7 Movable part of the furnace, opened
- 8 Plug for the insertion opening of the extractor device (electrode replacement)

Removing the tube



#### CAUTION

Risk of burns!

Allow the graphite tube furnace to cool down before removing the graphite tube.



#### NOTICE

Never touch the graphite tube with your bare fingers!

Fingerprints burn into the surface, and this causes premature damage to the pyrolysis coating of the tube.

1. Use the [OPEN FURNACE] button in the FURNACE / CONTROL window to open the graphite furnace (Fig. 42 on page 64).

- 2. Remove the graphite tube with tweezers. Use gloves when removing the tube by hand.
- 3. Insert the new graphite tube and use the [CLOSE FURNACE] button to close the furnace.

### 4.8.4 Formation of the graphite tube furnace

Formation of the graphite tube furnace will result in the following:

- atmospheric oxygen is expelled from the furnace
- the tube temperature is recalibrated
- the pyrolysis coating of the newly inserted graphite tube is conditioned
- the furnace is cleaned after pausing

It is recommended to form the furnace after the following procedures:

- after starting up the spectrometer
- after inserting a new graphite tube
- after closing the previously open furnace
- periodically after 50 to 100 measurements.

The complete formation program contains pre-programmed temperature stages.

Formation is started in the FURNACE / CONTROL window. During formation, the FORMAT TUBE window displays the current temperature stage, time and heating rate. During the first stage, the furnace and the graphite tube are cleaned and conditioned (contacts between the graphite tube and the electrodes are aligned).

- 1. Use the  $\bigcirc$  button in the ASpect LS to open the FURNACE / CONTROL window.
- 2. Enter the data for the current graphite tube in the FURNACE / CONTROL window:

New graphite tube	Heat cycles	0
	Life time	0
Used graphite tube	Heat cycles	Current value of the graphite tube
	Life time	Current value of the graphite tube

- 3. Click the [FORMATION] button.
  - ✓ The graphite tube can be used for measurements.

## 4.8.5 Cleaning / baking out the graphite tube

- 1. Use the  $\bigcirc$  button in the ASpect LS to open the FURNACE / CONTROL window.
- 2. Set the following parameters in the group box CLEAN FURNACE:

Темр. [℃]	End temperature to be reached during baking out. The final temperature should be at least 50 °C above the previous atomization temperature.
Ramp [°C/s]	Ramp
Hold [s]	Set the hold time

3. Use the [START] button to initiate the baking out process. If necessary, repeat the baking out process several times with higher temperatures.

# HydrEA method The following temper

The following temperature program must be used for baking out gold-coated and iridium-coated graphite tubes (also refer to the operating instructions for the hydride system). A higher final temperature is required to evaporate the metal coating.

	Baking out		Evaporation	
Element	Au	lr	Au	lr
Temp. [°C]	1000	2200	$1800 \le T \le 2600$	≤ 2600
Ramp [°C/s]	500s		500	
Hold [s]	10		10	
	Do not select a longer dwell time to avoid excessive load on the furnace			on the furnace.

Baking out or evaporation can be repeated several times.

# 4.9 Installing and adjusting the autosampler AS-GF

## 4.9.1 Installing the autosampler



## NOTICE

Always switch off the novAA 800 before installing or removing the AS-GF!

Connecting or disconnecting electrical contacts may damage the sensitive electronic components of the novAA 800.

Choose a safe location for the completion of the AS-GF. The device can tilt easily.



#### Fig. 44 Installed AS-GF

- 1 Left support in the sample chamber
- 2 Adjustment screw 1 (for Y coordinate)
- 3 Adjustment screw 2 (for X coordinate)
- 4 Tube holder
- 5 Tube guide with clamp nut
- 6 Adjustment screw 3 (for X coordinate)

1. Switch off the novAA 800.

- 7 Right support in the sample chamber
- 8 Purging position
- 9 Sample tray with cover
- 10 T valve of the dosing unit
- 11 Dosing syringe
- 12 Lock screw for piston rod
- 2. Install the tube guide (item 5 in Fig. 44) to the autosampler arm of the AS-GF and attach using the lock screw.

**Note:** The autosampler arm can be moved manually when the device is switched off.

- 3. Screw the dosing tube into the right opening of the T valve (item 10 in Fig. 44) on the dosing unit. Feed the dosing tube through the tube holder on the back of the autosampler and on the autosampler arm. Insert the dosing tube into the tube guide (item 5 in Fig. 44) until the tube end protrudes approx. 8 mm from the bottom of the tube guide; attach the tube using a clamp nut.
- 4. Plug the control line into the socket at the back of the AS-GF and screw it on.
- 5. Attach the depth-adjustable stop (item 4 in Fig. 39 on page 62) to the hinges on the sample chamber wall (item 5 in Fig. 39).

**Note:** The hinges that are used are the ones that are provided for the autosampler flame AS-F / AS-FD.

6. Unscrew the two plastic pins located next to the hinges of the AS-GF on the sample chamber wall and retain them for future use.

**Note:** The safety glass pane is attached to the plastic pins for the flame technique. The pins obstruct the attachment of the AS-GF.

- 7. Hang the AS-GF into the receptacles of the sample chamber (items 1 and 7 in Fig. 44). Check whether the autosampler is horizontally aligned. If necessary, adjust the position of the autosampler using the depth-adjustable stop.
- 8. If necessary, align the AS-GF with the furnace (coarse adjustment): Manually move the autosampler arm above the dosing opening of the graphite tube. If the dosing tube does not reach the opening, move the suspension of the autosampler backwards or forwards (in y-direction). Remove the autosampler from the sample chamber to do this. Use the adjustment screw 1 and the setscrew (2, 4 in Fig. 45) to move the left and the right suspension. The setscrews must be adjusted with a screwdriver. Fit the autosampler back on the suspension and check the coarse adjustment. Repeat the procedure as required.



Fig. 45 AS-GF with screws for aligning the furnace

Slider with left suspension mount

Slider with right suspension mount

Adjustment screw 1 2

1

- 3 4 Set screw
- 9. Plug the control cable into the socket on the connection panel of the AAS device (autosampler graphite connection, item 1 in Fig. 25 on page 49).
- 10. Place and fix the sample tray on the axis of the AS-GF.
- 11. Place the sample cover until it sits in the guide rail.
- 12. If applicable, mount the dosing syringe to the dosing unit ( $\rightarrow$  section "Replacing" the dosing syringe" on page 108).
- 13. Switch on the PC and the novAA 800, wait until the end of the spectrometer initialization, start the ASpect LS software and initialize the system.
  - The autosampler AS-GF is installed in the sample chamber.

Preparing the novAA 800 for the HydrEA technique

Prior to installing the HydrEA technique the graphite tube must be coated with iridium or gold (see hydride system manual). To do this, use the autosampler AS-GF with the dosing tube used during graphite operation. Alternatively, pipette the iridium or gold stock solution (c = 1 g/L) into the graphite tube by hand.

1. Use the autosampler to coat the graphite tube with iridium or gold.

# **1** Notice!

Do not use the titanium cannula for the coating.

- 2. Switch off the novAA 800 and install the hydride system (e.g. HS 60 modular).
- 3. For the HydrEA technique, loosen the clamp nut of the hose guide and pull out the dosing tube. Remove the dosing tube from the tube holder on the autosampler arm.
- 4. Insert the titanium tube into the hose guide until it protrudes from the bottom by 8 mm. Attach the titanium cannula with the clamp nut.
- 5. Attach the reaction gas tube (from the hydride system) to the titanium cannula.

Suitable autosamplers for the continuous introduction of samples into the hydride system HS 60 modular are the AS-F and the AS-FD.

#### 4.9.2 Adjusting the sampler

The AS-GF has already been installed in the sample chamber in accordance with section "Installing the autosampler" on page 67. The fine alignment of the AS-GF to the furnace is carried out in a software-controlled process. During this autosampler alignment, the dosing tube is put to the ideal position to deliver the samples in the graphite tube without touching the dosing insert, for instance. The injection depth for the sample is set in the same process.



Fig. 46 AS-GF adjusted

- 1 Adjustment screw 1 with lock nut
- 2 Clamp nut
- 3 Lock nut of adjustment screw 3
- 4 Adjustment screw 3
- 5 Adjusting aid with crosshair
- 6 Adjustment screw 2 with lock nut
- 1. Start the ASpect LS software, click on the symbol to open the AUTOSAMPLER window and switch to the TECHN. PARAMETERS tab.
- 2. Click the [ALIGN SAMPLER TO FURNACE] button to start the alignment.
- 3. Follow the prompts in the dialog fields of the software.

Align the AS-GF with the furnace:

- Pull the dosing tube from the tube guide of the autosampler by approx. 8 mm and fix it with a clamp nut.
- Replace the pipetting insert (dosing funnel) in the graphite furnace by the adjusting aid with crosshair.
- Use the buttons [UP]/[DOWN] to bring the autosampler arm to the height of the adjustment aid.
- Use the buttons [LEFT]/[RIGHT] to adjust the autosampler in x-direction (parallel to the optical axis) with the crosshair. The fine adjustment in x-direction must be done with the adjustment screws 2 and 3.
- Adjust the y-direction (sample chamber depth) using the adjustment screw 1.
- Tighten the screws and secure this setting with the lock nuts.
- Use the software to set the configuration in z-direction: Lower the autosampler arm up to the upper edge of the adjusting aid until the dosing tube just dips into the dosing opening.
- Click the [NEXT] button to save the settings for the x- and z-direction in the software.
- ✓ The autosampler arm returns to its initial position.
- Remove the adjusting aid and re-insert the dosing funnel.

Adjust the injection depth of the sample in the graphite tube:

- Loosen the clamp nut, place the dosing tube onto the tube bottom, check the position with the furnace camera, if necessary, and fasten the tube with the clamp nut.
- Use the software to configure the optimum injection depth of the autosampler arm above the bottom of the tube (approx. -0.8 mm for 20 µL pipetting volume).
- Click on [FINISH] to complete the adjustment process.
- ✓ The autosampler AS-GF is adjusted and ready for measurement.

Further configurations of the autosampler can be found in the "ASpect LS" manual in the section "Technical autosampler parameters".

#### 4.9.3 Loading the sample tray

1. Populate the positions of the AS-GF as follows:

Positions 1 – 100	1.5 mL sample cups
Positions 101 – 108	5 mL special cups

- 2. Place the sample cover with a tight fit.
- 3. Subsequent steps: Fill the purge bottle with purge solution (e.g. 1 % HNO<sub>3</sub>). If necessary, empty the waste bottle and dispose of the waste correctly.

**Note:** The population of the sample tray must match the software configuration in the method or in the sample ID.

## 4.9.4 Uninstalling the autosampler

- 1. Observe the sequence for switching off the novAA 800 and its accessories and turn the devices off.
- 2. For HydrEA coupling:

Remove the reaction gas tube from the titanium cannula. Loosen the clamp nut before pulling the titanium cannula off the tube guide.

- 3. Remove the control cable from the socket in the right side wall of the AAS device (autosampler graphite connection).
- 4. Loosen the adjustment screws 2 and 3 and remove the autosampler AS-GF from the sample chamber.
- 5. Remove the depth-adjustable stop from the sample chamber.
- 6. Screw the plastic pins back in.
### 4.10 Installing the flame technique



#### 4.10.1 Connections in the sample chamber for the flame technique

Fig. 47 Connections on the burner-nebulizer system

1 Burner

6

- 2 Markings for alignment on the mixing chamber tube 9 and the holding fixture 1(
- 3 Height adjustment
- 4 Suspension for SFS 6
- 5 Sample liquid supply
- 6 Outlet tube of the siphon
- 7 Connecting sockets for siphon sensor, injection module SFS 6 and scraper

- 8 Collection tray
- 9 Siphon
- 10 Oxidant connection (blue tube), auxiliary oxidant (black tube)
- 11 Fuel gas connection (red tube)
- 12 Attachment screw retaining clip
- 13 Stud bolt for fastening the burner
- 14 Automatic ignition unit



Fig. 48 Connections on the sample chamber walls

- 1 Suspension for injection module SFS 6
- 2 Suspension for autosampler, right side
- 3 Scraper connection

- 4 Connection for injection module SFS 6
- 5 Siphon monitoring connection
- 6 Suspension for autosampler, left side

#### 4.10.2 Software presettings for the flame technique

Set the option FLAME in the TECHNIQUE field of the QUICKSTART window of the ASpect LS software.

The user interface of the software is updated with the method and device parameters after initialization.

QUICK START 03.02.2021	17:27:32					
Instrument novAA 8	Instrument novAA 800D		ASpectLS Version: 1.7.0.0			analytikjena An Endress+Houser Company
OPERATOR: LAB.:	SuStein TecDoc			]		
TECHNIQUE:	Flame		~	-		
Worksheet		Last changed	Ву	Technique		<u>N</u>
Ag in strong matrix		03.04.2020 15:14	Analytik Jena	Graphite furnace (Wall)	Autosampler:	AS-FD
As in aqueous solu	As in aqueous solution (Hy)		Analytik Jena	Hydride	Sampler tray:	139 Pos.
As in aqueous solu	As in aqueous solution (HyEA)		Analytik Jena	HydrEA	Dumer.	<b>50</b> mm
Ba in strong matrix	Ba in strong matrix		Analytik Jena	Graphite furnace (Wall)		
Cu in aqueous solution		03.04.2020 14:56	Analytik Jena	Flame	Elements (Lar	np turret):
Cu in strong matrix		03.04.2020 15:15	Analytik Jena	Graphite furnace (Wall)	Cr;Mn;Fe;Co;I Na;K;Cr;Hg A	Nr;Cu Cd;Pb Dy u Ca;Tl;Pb;Bi Rh Mg
Hg in aqueous solu	Hg in aqueous solution (Hy)		Analytik Jena	Hydride		_
Hg in aqueous solu	ution (HyEA)	03.04.2020 15:02	Analytik Jena	HydrEA	~	
Favorites Recent	Predefined All		,C al	(12)		
					Sy	vstem check
Simulation						
				Skip Quick Start	Exit	ОК

Fig. 49 QUICKSTART window in the ASpect LS software

#### 4.10.3 Installation for manual sample introduction

With manual sample introduction the sample is loaded directly to the burner-nebulizer system. It is possible to use the injection module SFS 6.



#### NOTICE

Switch off the novAA 800 prior to installation! Connecting or disconnecting electrical plug-in contacts may damage the sensitive electronic components of the novAA 800.

- 1. Observe the sequence for switching off the novAA 800 and its accessories and turn the devices off.
- 2. Check that the mixing-chamber-nebulizer system properly sits in the retainer of the height adjustment. The plate of the mixing chamber tube must have contact. The white marking on the mixing chamber tube must be positioned above the edge of the retainer (item 2 in Fig. 50).



Fig. 50 Flame technique, manual sample introduction

- 1 Burner
- 2 Markings for the burner alignment on the mixing chamber tube and the retainer
- 3 Mixing-chamber-nebulizer system
- 4 Sample aspiration hose on the nebulizer
- 5 Siphon sensor connection line
- 6 Collection tray

- 7 Outlet tube from the siphon
- 8 Sample cup
- 9 Oxidant connection (blue), auxiliary oxidant (black) and fuel gas (red)
- 10 Retainer for the height adjustment
- 1. Attach the outlet tube to the connector of the siphon and the corresponding opening in the lid of the waste bottle.

**Note:** Route the outlet tube in a constant downward slope. If necessary, shorten the tube. The tube must not dip into the liquid.

- 2. Fill the siphon with water via the mixing chamber tube until water flows out via the outlet tube.
- 3. Plug the connector of the siphon sensor to the connection on the right sample chamber wall (item 5 in Fig. 48 on page 73).
- 4. Push the collection tray underneath the burner-nebulizer system in the sample chamber.
- 5. Connecting the gas supply:
  - Fuel gas hose (red) at the top of the mixing chamber head
  - Oxidant hose (blue) at the side of the nebulizer
  - Auxiliary oxidant hose (black) at the side of the mixing chamber

Only tighten the knurled-head screws finger tight.

- 6. Plug the nebulizer into the mixing chamber head and lock it with the ring.
- 7. Attach the aspiration tube to the nebulizer cannula.

	<ol> <li>Attach the required burner (50 mm or 100 mm) to the mixing chamber tube, turn to the stop position and clamp. Ensure that the burner is positioned correctly. There are markings on the mixing chamber tube and the retainer.</li> </ol>					
	9. Injection module SFS 6 If you are working with the injection module SFS 6, install the injection module SFS 6 ( $\rightarrow$ section "Installing the injection module SFS 6" on page 80).					
	10. Place the sample or purge cups in front of the device or on a separate side table.					
	11. Hang the safety glass in and slide it in front of the burner.					
	12. Switch on the novAA 800 and start the ASpect LS software.					
	✓ The burner-nebulizer system is installed and prepared for manual sample in- troduction.					
Steps	1. Observe the sequence for switching off the novAA 800 and turn the device off.					
Demounting	<ol> <li>If you worked with the injection module SFS 6, put the injection module SFS 6 out of operation (→section "Installing the injection module SFS 6" on page 80).</li> </ol>					

3. Remove sample cups and purge cups.

#### 4.10.4 Installation for continuous operation / sample introduction via autosampler

novAA 9 1 2 8 3 7 Î 4 5 6

In continuous working mode, the samples are loaded via the autosampler AS-F or AS-FD.

Flame mode, continuous with autosamplers AS-FD and SFS 6 Fig. 51

- Storage bottle for diluent 1
- 2 Fluidics module with dosing unit
- 3 Storage bottle for washing liquid
- Tube for washing liquid to the SFS 6 4
- 5 Coated tubes for washing liquid and diluent (towards the AS-FD)
- 6 Autosampler AS-FD with sample tray
- 7 Injection module SFS 6 (where applicable)
- 8 Sample intake tube
- Tube for diluent (thick cannula) and sample 9 intake tube (thin cannula)

1. Observe the sequence for switching off the novAA 800 and its accessories and turn Installing the burnerthe devices off. nebulizer system 2. Check that the mixing-chamber-nebulizer system properly sits in the retainer of the height adjustment. The plate of the mixing chamber tube must have contact. The mixing chamber must be aligned to the height adjustment, the marking on the connector must be above the edge of the holding fixture (item 2 in Fig. 50 on page 75). 3. Attach the waste tube to the connector of the siphon and the corresponding opening in the lid of the waste bottle. **Note:** Route the outlet tube in a constant downward slope. If necessary shorten the tube. The tube must not dip into the liquid. 4. Fill the siphon with water via the mixing chamber tube until water flows out via the waste hose. 5. Plug the connector of the siphon sensor to the connection on the right sample chamber wall (item 5 in Fig. 48 on page 73). 6. Push the collection tray underneath the burner-nebulizer system in the sample chamber. 7. Connecting the gas supply: Fuel gas hose (red) at the top of the mixing chamber head Oxidant hose (blue) at the side of the nebulizer Auxiliary oxidant hose (black) at the side of the mixing chamber Only tighten the knurled-head screws finger tight. 8. Plug the nebulizer into the mixing chamber head and lock it with the ring. 9. Attach the required burner (50 mm or 100 mm) to the mixing chamber tube, turn to the stop position and clamp. Ensure that the burner is positioned correctly. There are markings on the mixing chamber tube and the retainer. Installing the If you are working with injection module SFS 6, install injection module SFS 6 (section injection module "Installing the injection module SFS 6" on page 80). Installing the 1. Hang the autosampler in the corresponding receptacles of the sample chamber (items 2, 6 in Fig. 48 on page 73). Adjust the adjustment screw on the right susautosampler pension so that the autosampler cannot slip out of the mounting hole (item 3 in Fig. 52 on page 78). 2. Place the Fluidics module (for AS-FD) or storage bottle for washing liquid (for AS-F) next to the AAS device. 3. Plug the control cables for connecting the autosampler to the Fluidics module and the AAS device into the connections on the rear of the autosampler and lock them in place (items 1 and 2 in Fig. 52 on page 78). If necessary, unhinge the right side of the autosampler to do this. 4. Plug the control cable into the "Sampler flame" connection on the right side wall of the novAA 800 (item 2 in Fig. 25 on page 49) and lock it in place. 5. Attach the outlet tube to the outlet connector of the autosampler (rear panel, item 4 in Fig. 52 on page 78). Attach the outlet tube to the respective opening of the lid of the collecting bottle.

**Note:** Route the outlet tube in a constant downward slope. If necessary shorten the tube. Tube must not dip in the liquid.

6. Screw the tube for the purging liquid into the rear of the autosampler (item 5 in Fig. 52 on page 78).

**Note:** In the AS-FD the tubes for connecting the autosampler with the Fluidics module are numbered and attached to each other by a jacket. The tubes are attached to the rear of the autosampler using the attachment lug. Marking Wash tube "2".

7. Feed the dosing tube for the diluent (marking "1") in the AS-FD through the tube guide on the autosampler arm and attach it to the thicker cannula of the autosampler arm.

**Note:** The autosampler arm can be moved manually when the device is switched off.

- 8. Stick the sample aspiration tube onto the nebulizer cannula.
- 9. Route the sample intake tube through the tube guide at the autosampler arm and plug it onto the thin cannula of the autosampler arm.
- 10. Lay the sample tray on the autosampler casing, make sure it clicks into place.
- 11. Place the sample cover until it sits in the guide rail.



Fig. 52 Rear view of the autosampler AS-FD

- 1 Fluidics module connection
- 2 AAS connection
- 3 Suspension mount with adjustment screw
- 4 Connector for outlet tube
- 5 Screw for purge tube
- 6 Suspension for injection module SFS 6

Preparing the Fluidics module (for AS-FD)



- 1 Storage bottle for purging liquid
- 2 Diluent connection
- 3 Dosing tube connection (to AS-FD)
- 4 Dosing syringe, consisting of piston and glass cylinder
- 5 Drive rod with attachment screw
- 6 Storage bottle for diluent

- Fig. 53 Dosing unit at the Fluidics module of the AS-FD
- 12. If applicable, mount the dosing syringe to the dosing unit ( $\rightarrow$  section "Replacing the dosing syringe" on page 108).
- 13. Place the storage bottles for the washing liquid (left) and diluent (right) into the bottle holders of the Fluidics module.
- 14. Immerse the short tube (marking at the tube "3") into the storage bottle for the diluent. Screw the second tube end to the valve (item 2 in Fig. 53).
- 15. Screw the dosing tube for the diluent (sheathed, marking "1") to the second connection of the valve (item 3 in Fig. 53).

16. Immerse the hose for the washing liquid (marking "2") into the storage bottle.

Uninstalling the autosampler

- 1. Observe the sequence for switching off the novAA 800 and turn the device off.
- 2. Detach the sample aspiration tube from the thin cannula of the autosampler arm and pull it off the tube guide.
- 3. Detach the tube for the wash liquid at the rear of the autosampler.
- 4. For the AS-FD, detach the dosing tube for the diluent from the thicker cannula and pull it off the tube guide. Pull the two encased tubes out of the attachment lug at the rear of the autosampler.
- 5. Pull the outlet tube from the connector of the autosampler (backplate).
- 6. Detach both control cables at the rear of the autosampler.
- 7. Take the autosampler out of the sample chamber.

Uninstalling theIf you are working with the injection module, put the injection module SFS 6 out ofinjection moduleoperation ( $\rightarrow$  section "Uninstalling " on page 81).

#### 4.10.5 Installing the injection module SFS 6

- 1. Screw on the aspiration tubes to the injection module:
  - Medium length tube into the upper connection to the sample (item 1 in Fig. 54)
  - Short tube into the lateral connection to the nebulizer cannula (3)
  - Long tube into the bottom connection to the washing liquid (4)
- Manual working mode: Attach the injection module to the suspension on height adjustment.
   Work with an autosampler: Attach the injection module to the holder of the autosampler (item 6 in Fig. 52 on page 78).
- 3. Insert the control cable (item 5 in Fig. 54) into the central connector on the right side of the sample chamber wall.
- 4. Fit the short tube (3) to the nebulizer cannula.
- 5. Dip the long tube (4) into the storage bottle with the washing solution.
- 6. Dip the medium-length tube (1) into the sample cup or connect it with the aspiration cannula of the autosampler.
  - ✓ The injection module SFS 6 is ready for measurements.



Fig. 54 SFS 6 for manual sample introduction, installed

- 1 Tube to the sample / autosampler
- 2 Injection module SFS 6
- 3 Tube to the nebulizer

- 4 Tube to the purge solution
- 5 Connecting cable for the control of the SFS 6

Installing the injection module SFS 6

Uninstalling the injection1.Remove the aspiration tubes from the washing liquid bottle and the sample cup<br/>(for manual operation), or pull them off the aspiration cannula of the autosampler<br/>to allow the system to drain.

- 2. Pull off the short piece of tube from the nebulizer cannula.
- 3. Detach the control cable of the SFS 6 from the AAS, remove the injection module.

#### 4.10.6 Replacing the burner



#### CAUTION

**Risk of burns!** 

To remove the hot burner, use the burner bracket (optional accessory). Otherwise wait until the burner has cooled down.

- 1. Push the safety glass pane upwards.
- 2. Loosen the lock screw of the burner and take the burner off. If available, use a burner fork.
- 3. Place the new burner on the mixing chamber tube, turn against the 0°-stop and fasten it with the fixing screw. Ensure that the burner is positioned correctly. There are markings on the mixing chamber tube and the retainer.
  - ✓ The new burner is fully installed.

#### 4.10.7 Installing the scraper

When working with the nitrous oxide flame the use of a scraper is recommended which automatically removes carbon deposits from the burner head in nitrous oxide operation. Alternatively, carbon deposits can be manually removed from the burner slot with the scraper. Upon request, the scraper can be mounted to the 50-mm burner in the factory before delivery. It can also be retrofitted on a 50 mm burner.



#### NOTICE

For fuel gas flows > 250 NL/h, pay attention to stubborn deposits. Remove these where necessary to ensure the functionality of the scraper.

- 1. Unscrew the screws from the front burner jaw (arrow in Fig. 55) (the screw for fastening the burner on the mixing chamber tube is also located on the side of the front burner jaw).
- 2. Unscrew the fastening rail (item 1 in Fig. 56) with the knurled head screws (item 3 in Fig. 56) from the scraper.

The captive knurled head screws remain attached in their holder in the scraper.

3. Mount the fastening rail to the burner body as shown in Fig. 56. Use the three longer titanium screws and the nuts that were supplied with the equipment to mount the rail. Insert the screws from the top through the front burner jaw and screw down the fastening rail with nuts.

4. Attach the scraper to the guide pins of the fastening rail (item 2 in Fig. 56) and tighten it with knurled head screws (item 3 in Fig. 56).



Fig. 55 Screws on the front burner jaw



Fig. 56 Fastening rail / knurled head screws on the scraper

- 3 Knurled head screws
- 2 Guide pins

1

Fastening rail for the scraper

### 4.11 Starting up the novAA 800 with accessories

#### 4.11.1 Switching on sequence

- 1. Switch on the exhaust unit.
- 2. Switch on the PC and wait for the operating system to initialize: The application icons appear on the screen, including the ASpect LS program icon.
- 3. Switching on the novAA 800: Press the green ON/OFF switch on the right side wall. Wait until the spectrometer has fully completed the automatic initialization routine.
- 4. Start ASpect LS: Double-click with the mouse cursor on the ASpect LS icon.
- 5. Connect the printer and the compressor if they are needed.
  - ✓ The AAS system is now switched on, work (analysis preparation and measurement) may begin.



#### NOTICE

The mobile cooling unit KM5 is controlled by the novAA 800 and is therefore not switched on and off manually.

#### 4.11.2 Switching off sequence

- 1. Close the control software ASpect LS on the PC: Click the menu item FILE > EXIT.
- 2. For unsaved values decide whether data or information should be saved before exiting the program.
- 3. Shut down the PC.
- 4. Use the respective mains switches to switch off (in this order):
  - Compressor
  - AAS accessories (e.g. hydride system)
  - novAA 800
  - Printer
  - PC
  - ✓ The AAS system is now switched off.

# 5 Service and maintenance



#### WARNING

Electric shock! The novAA 800 must be switched off before carrying out any maintenance work. Pull the mains plug. The safe disconnection of the novAA 800 from the mains can only be achieved by pulling the mains plug. Power is still supplied to both certain areas of the spectrometer, as well as the output socket, after the device has been switched off at the main switch.

The only exception to this are maintenance tasks that must be carried out while the AAS device and the control software are running, such as the baking out of the graphite tube.



#### WARNING

Risk of skin and eye damage caused by UV radiation!

HCL,  $D_2$ -HCL, the heated graphite tube (T > 1000 °C) and the flame of the burner transmit radiation in the UV range. Do not look into the rays emitted by the lamp, the graphite tube or the flame without UV protection goggles. Protect the skin against radiation.

Switch off the lamp by means of the control and analysis software ASpect LS before opening the lamp door: To do so, open the section OPTICAL PARAMETERS in the SPECTROMETER / CONTROL window of the ASpect LS software and set the lamp current [mA] to zero. Open the drop-down list BACKGROUND CORRECTION and select the option NO BACKGROUND. Click [SET]. Negate the error message.

To observe the placement of the samples or the drying of liquid samples, the dental mirror may only be inserted into the beam path from the left side of the graphite furnace. When observing on the right side of the furnace, UV radiation may be reflected.



### WARNING

The operator is responsible for ensuring the proper decontamination of the device before maintenance and repair works. This applies to any case in which the inside or the outside of the device have been contaminated with hazardous substances.



#### CAUTION

The operator must not undertake any service or maintenance work to this device and its components other than those specified and described in this chapter.

Please observe the notes in section "Safety instructions" on page 9. 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 and analysis software ASpect LS.

To guarantee faultless and safe functioning, the novAA 800 should be inspected on an annual basis by the Analytik Jena service department.



#### CAUTION

Risk of burns from touching hot surfaces! Observe the required cooling times before starting any maintenance tasks on the graphite furnace and the burner-nebulizer system.

## 5.1 Maintenance overview

Maintenance item	Action	Frequency
Basic unit		
Fuse	Change the fuse.	As required.
Sample chamber	Clean. Remove fluid from the collection	Regularly. If there are residues in the tray.
	Clean the windows for beam entry and exit in the sample chamber	in case of visible contamination such as streaks and burnt-in residues, or
Fans (rear panel)	Check the ventilation grilles for con- tamination and clean them, if re- quired.	Monthly.
Gas connectors	Check for leaks.	When connections are newly connected and if the manometer of the external gas supply shows a significant loss of pressure.
Atomization unit	Align within the beam path.	novAA 800 G + F: automatic height adjust- ment, depth is factory preset. novAA 800 D: automatic height adjustment, depth of the flame atomizer can be adjusted manually by means of an adjustment screw.
Graphite tube furnace		
Furnace window	Wipe with a lint-free cloth soaked in alcohol.	Daily or weekly, depending on the sample ma- trix.
	Clean with a mild detergent.	In case of stubborn contaminations.
Graphite surfaces	Wipe the contact surfaces of the electrode in the movable part of the furnace with a lint-free cloth soaked in alcohol or clean them with a cot- ton swab.	Daily.
Graphite tube	Clean using the computer-controlled baking out program.	Daily.
	Replace.	In case of significant burn-up, a strong decline in sensitivity and very high RSD% values.
Iridium-coated or gold-coated graphite tube	Evaporate the metal layer.	After approx. 500 atomizations or for a new coating (malfunctions will lead to distorted measurement results).
Electrodes and furnace shroud	Clean contact surfaces of the elec- trodes with a cotton swab, a clean cloth soaked in alcohol, or nonwo- ven paper. Check for wear,	Daily or weekly, when working with matrix modifiers (MgNO <sub>3</sub> ) immediately after the ap- plication Monthly,
	replace if necessary.	as required.
Pipetting insert	Clean and wash.	May be necessary on a daily basis, depending on the type of samples.

Maintenance item Action		Frequency		
Burner-nebulizer system				
Burner-nebulizer system	Disassemble and clean, optimize sensitivity if necessary.	Depending on analyzed sample material; biological samples or samples with a high salt content require more frequent cleaning.		
Sensor for burner detection	Clean with alcohol.	In case of visible contamination or if the mounted burner is not recognized by the soft- ware.		
Injection module SFS 6	Check hoses for deposits, kinks and cracks, replace if necessary.	Regular checks, replace hoses as required.		
Autosampler AS-GF, AS-F and AS	-FD			
Dosing tube / SEP cannulas	Check for deposits, kinks and cracks, replace if necessary.	Regular checks because deposits can distort the measurement results.		
Purge cups,	Clean.	Regularly.		
mixing cups	Check that the purge cups are non- porous and free of blisters.	Regularly, especially after filling.		
Dosing syringe in the dosing unit	Replace.	As required (in case of leaks).		
Cooling unit KM 5				
Water tank	Check the level and cleanliness of the cooling liquid.	Quarterly.		
	Fill and vent the tank.	If necessary.		
	Clean the tank.	lf necessary.		
Maintenance item	Action	Frequency		
Piston compressor PLANET L-S50	-15			
Pressure cylinder, fluid separator on the pressure reducer	Drain condensation water.	Weekly.		
Suction filter	Check.	Monthly.		
	Clean, replace if necessary.	Every six months.		
Oil	Check the oil level.	Weekly.		
	Change the oil.	Annually		

#### Maintaining the basic device 5.2

#### 5.2.1 **Replacing the fuses**



#### WARNING

**Risk of electric shock!** 

Prior to replacing the fuses in the novAA 800 always switch off the device with the mains switch and pull the plug.

The power supply fuses (F1, F2) of the novAA 800 D and G may only be replaced by Analytik Jena service engineers or by technical personnel authorized by Analytik Jena.

The fuses of the novAA 800 D and G can be found on the rear panel of the device and novAA 800 D + G within the sample chamber. They are marked.

Fuses

Def -. ~ 7 го ( ι. fuses

rear panel

Refer	to	Fig.	27	on	page	50	tor	the	tı

Fuse number	Туре	Protected circuit
F3	T 6.3 A/H	Socket for external accessories
F4	T 6.3 A/H	Socket for external accessories
F5	T 2.5 A/H	Transformer, primary side, NTL
F6	T 2.5 A/H	Transformer, primary side, NTL
F7	T 0.08 A	D <sub>2</sub> -HCL
F8	T 0.25 A	HCLs
F9	T 3.15 A	Filament

Fuse for the furnace

Refer to item 7 in Fig. 40 on page 63 for the furnace fuse

Туре	Protected circuit
TR5-T 100 mA	Measuring lead, graphite tube furnace

novAA 800 F

The fuses of the novAA 800 F can be found on the rear side of the device (Fig. 27 on page 50).

Fuse number	Туре	Protected circuit
F1	T 6.3 A/H	Socket for external accessories
F2	T 6.3 A/H	Socket for external accessories
F3	T 2.5 A/H	Transformer on the primary side, SNT
F4	T 2.5 A/H	Transformer on the primary side, SNT
F5	T 0.08 A	D <sub>2</sub> -HCL
F6	T 0.25 A	HCLs
F8	T 3.15 A	Filament

#### 5.2.2 Cleaning the sample chamber

- 1. Clean the sample chamber regularly using a lint-free cloth moistened with alcohol.
- 2. In case of residual fluid in the collection tray underneath the mixing-chambernebulizer system (e.g. from the siphon outlet), carefully remove and empty the tray and rinse it under the tap.
- 3. Clean the radiation entrance and exit windows of the sample chambers if energy losses are detected:
  - Turn the windows to unscrew them from the bayonet lock.

#### 1 Notice!

Do not clean the furnace windows in an ultrasonic bath. This may lower the UV permeability of the windows.

- Wipe the windows with a lint-free cloth soaked in alcohol (optical cloth) without leaving streaks.
- Re-insert the windows to the sample chamber.

**Note:** After cleaning the window with alcohol, it takes about 1 h before the UV transmission is completely restored.

#### 5.2.3 Checking the gas connections for leaks

The gas connectors (on the rear panel of the device) must be checked for leaks:

- Weekly in form of a safety check.
- If a gas connection is opened when operation is started up again.

To check for leaks, close the stopcock of the gas supply system and observe the pressure reading on the downstream manometer. If the pressure drops significantly, search and fix the gas leak using the following method:

- 1. Brush connections with a heavily foaming liquid (e.g., soap solution). If bubbles form when opening the gas connection, switch off the novAA 800 and disconnect the gas supply.
- 2. Unscrew the untight gas connections and check whether they are properly positioned. Replace worn out sealing rings. Cut off worn out hose ends.
- 3. Screw on the gas connectors by hand or using a suitable open-end wrench. When doing so, make sure that the connector is properly placed on the port.
- 4. Re-check the gas connections for leaks.

### 5.3 Graphite tube furnace

After a prolonged operation time, sample residues, modifiers and sublimated carbon from the graphite tube are deposited on the contact surfaces of the electrodes, the furnace shroud and the pipetting insert. Such deposits may lead to deviations in the effective tube temperature and contaminate the samples.

Damages to furnace, ceramic ring, graphite tube or electrodes may be the reason for poor analysis results, too.



#### CAUTION

Risk of burning at the hot furnace!

Allow the graphite tube furnace to cool down before attempting any service or maintenance work.

#### 5.3.1 Cleaning the furnace windows



#### NOTICE

Do not touch the quartz panes of the furnace windows with your bare hands. Fingerprints burn in.

Do not clean the furnace windows in an ultrasonic bath. This may lower the UV permeability of the windows.

Danger of embrittlement of the rubber seals. When cleaning the furnace windows with a cloth soaked in alcohol, make sure that the rubber seals do not come in contact with the alcohol!

The furnace windows must be cleaned every week with a lint-free cloth soaked in alcohol (optical cloth) without leaving streaks. **Note:** After cleaning the furnace window with alcohol, it takes about 1 h before the UV transmission is completely restored.

Use a mild surfactant to remove stubborn soiling. Preparing the cleaning solution: Use a mixture of demineralized water and 1 vol.% cleaning solution.

- 1. Pull off the furnace windows by hand in a rotating movement. Do not touch the windows!
- 2. Fill so much cleaning solution into a beaker that the furnace windows can be completely submerged in the solution.
- 3. Let the windows soak in the solution for approx. 30 min at a temperature between 25 and 30 °C.
- 4. Remove the furnace windows from the cleaning solution (e.g. using plastic pliers, do not hold the window by the optical surfaces) and rinse them with demineralized water ( $\sigma < 1 \ \mu$ S/cm).
- 5. Use compressed air or argon to blow dry the windows.
- 6. Re-insert the furnace windows. Identical markings must point upwards ( $\rightarrow$  Fig. 57 on page 90)!

If the furnace windows are too loose or if the sealing rings of the furnace windows exhibit brittleness and cracks, replace the sealing rings.

✓ The furnace windows are cleaned and re-installed.



Fig. 57 Markings on the furnace windows

#### 5.3.2 Cleaning the graphite surfaces

The graphite surfaces must be cleaned every day after using the device.

- 1. Switch on the novAA 800 and start the ASpect LS software (the movable furnace part must be pressurized to be opened/closed).
- 2. Use the  $\bigcirc$  button in the ASpect LS to open the FURNACE window. Open the CONTROL tab.
- 3. Use the [OPEN FURNACE] button to open the furnace.
- 4. Remove the pipetting insert from the furnace shroud and clean it in 0.1 to 1 modular HNO<sub>3</sub>. Then rinse well with slightly acidic or demineralized water.
- 5. Clean the contact surfaces of the electrode on the movable part of the furnace with a cotton swab, a lint-free cloth soaked in alcohol, or nonwoven paper.
- 6. Clean inner surfaces of the furnace shroud with a cotton swab.
- 7. Use the [CLOSE FURNACE] button to close the graphite furnace.
  - ✓ The graphite furnace is ready for operation again.

#### 5.3.3 Cleaning and changing the graphite tube

Clean the graphite tube	<ul> <li>Bake out the graphite tube every day to clean it.</li> </ul>			
	Refer to chapter "Cleaning / baking out the graphite tube" on page 67 for the required steps.			
Cleaning the coated	<ul> <li>For the HydrEA technique, bake out the graphite tube every day to clean it.</li> </ul>			
graphite tube	Refer to chapter "Cleaning / baking out the graphite tube" on page 67 for the required steps.			
Evaporating the iridium coating	<ul> <li>Evaporate the iridium or gold coating after approx. 500 atomizations or before recoating.</li> </ul>			
	Refer to chapter "Cleaning / baking out the graphite tube" on page 67 for the required steps.			
Replacing the graphite tube	The graphite tube must be replaced if it shows significant burn-up or if it no longer meets analytical requirements. In that case, the pyrolysis layer is run down.			

Refer to chapter "Inserting the graphite tube into the furnace" on page 64 for the required steps.

#### 5.3.4 Replacing electrodes and furnace shroud

Electrodes and furnace shroud must be replaced if the device persistently yields poor analytical results which cannot be adjusted by cleaning or replacing the graphite tube.



3 Electrodes
 Furnace shroud

Fig. 58 Electrodes and graphite tube sheath

These tasks can be completed by the service department in the course of the regular maintenance. If you wish to perform the maintenance work yourself, you will need the furnace tools that are available as an option.



- 1 Insertion tool for furnace shroud
- 2 Extractor
- 3 Insertion tool for
- electrodes
- 4 Allen wrench
- 5 Ratchet wrench
- 6 Key wrench

Fig. 59 Furnace tools



#### NOTICE

The graphite furnace shown in the following photo series has been removed from the device to ensure a good view on the individual work steps. However, the graphite furnace does not need to be removed from the sample chamber of the novAA 800 to perform this maintenance work.

- 1. Switch on the novAA 800 and start the Aspect LS software (the movable furnace part must be pressurized to be opened/closed).
- 2. Use the  $\bigcirc$  button in the ASpect LS graphite furnace technique to open the

FURNACE / CONTROL window.

- 3. Use the [OPEN FURNACE] button to open the furnace.
- 4. Use a pair of tweezers to remove the graphite tube from the opened graphite furnace. Wear gloves when removing the tube by hand.



5. Unscrew the cover screw from the movable part of the furnace.



6. Withdraw the retaining pin from the movable part of the furnace and fold the movable part of the furnace downwards.



7. Carefully loosen the insulating ring with the key wrench and unscrew it completely by hand.

**Risk of breaking the insulating ring!** Do not tilt the key wrench!



8. Screw the extractor with the spindle turned back into the movable part of the furnace as far as it will enter.

Use the ratchet wrench to fully push out the electrode.

Remove the extractor from the part of the furnace.

9. Pull the furnace window off the furnace shroud. Remove the pipetting insert.

10. Detach the three gas hoses. To do this, push in the ring on the quick release and pull out the hose.

Use the Allen key to carefully unscrew the three gas connectors. To do this, insert the Allen key into the gas connectors and turn the key counterclockwise.



T



Make sure that the electrode and the furnace part are parallel when positioning and inserting the electrode. If the electrode is jammed, remove the electrode completely and start again.



- 16. Align the furnace shroud with the cylindrical receptacle parallel to the furnace body and fasten it with the inserting tool (large bracket).
- 17. Insert the furnace shroud to the stop. Loosen the insertion tool and remove it.

**Risk of breaking the furnace shroud!** While inserting the furnace shroud make sure that it is parallel to the stationary part of the furnace. If the furnace shroud gets jammed, press it out completely and start again.

- 18. Screw the sensor sleeve for the temperature sensor for the cooling water finger-tight onto the screws of the stationary part of the furnace.
- 19. Insert the sensor to the sensor sleeve and tighten it with the union nut.

20. Check the sealing rings of all three gas connectors and replace if damaged.



21. Screw the gas connector for the outer gas flow finger-tight into the stationary part of the furnace transversely from below.

Attach the white gas hose to the gas connector.









22. Screw the two other gas connectors for the inner gas flow to both ends of the furnace shroud.

Attach the two gas hoses to the gas connectors.

23. Position a new electrode parallel to the movable part of the furnace and fix it with the insertion tool (small bracket).

Insert the electrode as far as it will go into the jaw of the furnace using the ratchet wrench.

**Risk of breaking the electrode!** Make sure that the electrode does not get jammed.

Remove by suction or blow away any graphite dust which is present.

24. Attach the furnace window to the furnace shroud. Insert the pipetting insert.

**Note:** The identical markings on the furnace window must face upwards (see Fig. 57 on page 90).

25. Screw in the insulating ring by hand before moderately tightening it as far as it goes using the key wrench.

**Risk of breaking the insulating ring!** Do not tilt the key wrench!

26. Insert the retaining pin into the furnace jaw and the connecting rod (arrow) as far as it goes. The connecting rod must be in the front position.



27. Screw the cover screw into the movable part of the furnace.

28. Close the furnace with the [CLOSE FURNACE] button.

✓ Electrodes and furnace shroud are fully installed into the graphite furnace.

Insert the graphite tube into the furnace before re-starting the furnace ( $\rightarrow$  section "Inserting the graphite tube into the furnace" on page 64). Form the graphite tube.

#### 5.4 Burner-nebulizer system

The burner-nebulizer system must be cleaned at regular intervals. The following conditions indicate when the system needs to be cleaned:

- Irregularities in the flame hem of the burner flame. Washing with diluted acid in the active program and blowing the burner out does not bring about any improvement.
- The sensitivity given in the cookbook for an individual element is not achieved despite changing the composition of the gas.
- Hard deposits on the burner slit occurring during the analysis of solutions with a high salt content cannot be removed with the cleaning strip.



#### CAUTION

Risk of burns! Allow the burner to cool down before attempting any service or maintenance work.

Undertake the following maintenance work to the burner-nebulizer system:

- 1. Take the burner-nebulizer system apart.
- 2. Clean the burner.
- 3. Clean the nebulizer.
- 4. Clean the siphon.
- 5. Clean the mixing chamber.
- 6. Assemble the burner-nebulizer system.
- 7. Adjust the sensitivity of the burner-nebulizer system (optimize flame).

### 5.4.1 Taking the burner-nebulizer system apart



Fig. 60 Removing and disassembling the burner-nebulizer system

- 1 Burner
- 2 Retaining screw on the burner
- 3 Mixing chamber tube
- 4 Locking ring for the nebulizer
- 5 Mixing chamber screw joints (4 pieces)
- 6 Nebulizer
- 7 Outlet tube on the siphon

- 8 Connection for the siphon sensor
- 9 Siphon sensor
- 10 Screwed tube connections on the mixing chamber head and the nebulizer
- 11 Screwed tube connections on the mixing chamber head
- 12 Knurled head screw on the retaining clip



Fig. 61 Mixing chamber and nebulizer disassembled for cleaning

- 1 Safety plug
- 2 Mixing chamber tube
- 3 Impeller
- 4 Mixing chamber head with connections for gases, nebulizer and siphon
- 5 Connections for auxiliary oxidant and fuel gas (pointing backwards)
- 6 Nebulizer connection with locking ring
- 7 Baffle ball
- 8 Nebulizer with connection for oxidant and connection for sample tube
- 9 Clamp screw
- 10 Siphon
- 11 Siphon sensor



Fig. 62 Withdrawing the nebulizer from the mixing chamber

- 1. Loosen the retaining screw (item 2 in Fig. 60 on page 98) on the burner and remove the burner from the connector.
- 2. Unscrew the screwed tube connections on the mixing chamber head and the nebulizer (items 10 and 11 in Fig. 60) and pull the sample uptake tube off the nebulizer.
- 3. Turn the locking ring of the nebulizer (item 4 in Fig. 60) to open the locking. Withdraw the nebulizer from the mixing chamber head, holding the nebulizer in the groove (Fig. 62).

#### Risk of breaking the connector!

Connector for gas connection may break when being pulled.

- 4. Unscrew the siphon sensor cable from the connection in the sample chamber (item 8 in Fig. 60) and pull it off.
- 5. Remove the outlet tube from the outlet connector of the siphon (item 7 in Fig. 60).
- 6. Loosen the clamp screw of the siphon and pull off the siphon towards the bottom. Empty the siphon.

#### 🗥 Caution

The solution in the siphon is acidic. Wear protective goggles and clothing.

- 7. Unscrew the insert of the siphon sensor and remove the sensor from the siphon (item 11 in Fig. 61).
- 8. Hold the system tightly, loosen the knurled head screw on the holding bow of the mixing chamber tube (item 12 in Fig. 60), push the retaining clip backwards and remove the system.
- 9. Withdraw the safety plug (item 1 in Fig. 61) from the mixing chamber.
- 10. Loosen the four screw joints of the mixing chamber (item 5 in Fig. 60) and disassemble the mixing chamber into the chamber head and the chamber tube.
- 11. Remove the impeller (item 3 in Fig. 61) from the chamber tube.
- 12. Unscrew the gas connections for fuel gas and auxiliary oxidant (item 5 in Fig. 61) from the mixing chamber head.

#### 5.4.2 Cleaning the burner

- 1. Clean the burner under running water.
- 2. Clean the burner with the burner jaws facing downwards in an ultrasonic bath for 5 10 min with diluted HNO<sub>3</sub> (c = 0.1 mol/L). If there is no ultrasonic bath: Place the burner overnight in diluted HNO<sub>3</sub>.

Do not use hydrochloric or hydrofluoric acid as they might damage the burner!

3. Rinse the burner with distilled water. Let the burner dry.

Removing hard deposits Perform the following working steps only when hard deposits haven't been removed by the procedure described above.

- 1. Undo the srew joints (item 2 in Fig. 63) of the burner jaws on the burner body and remove the burner jaws.
- 2. Remove the burnt residue deposits with burner cleaner (paper strips).
- 3. Clean the burner jaws in 0.1 molar HNO<sub>3</sub>, and then wash with distilled water.
- 4. Screw the burner jaws onto the burner body. The dowel pins (item 3 in Fig. 63) on the burner ensure correct positioning.



Fig. 63 Screw joints of the burner

- 1 Juxtaposing screw joints of the burner (Do not loosen the screws)
- 2 Screw joints attaching the burner jaws to the burner body
- 3 Dowel pins on the bottom side of the burner jaws

#### 5.4.3 Cleaning the nebulizer

- 1. Place the nebulizer for several minutes into an ultrasonic bath with approx. 1 % of HNO<sub>3</sub> or organic solvent (isopropyl alcohol).
- 2. Slightly turn the baffle ball (item 7 in Fig. 61 on page 98) and pull it off the nebulizer. If the baffle ball is seized, place the nebulizer back into the ultrasonic bath for another few minutes.
- 3. Insert the cleaning wire into the nebulizer canula and clean the canula by moving it up and down several times.
- 4. Attach the baffle ball on the nebulizer and lock it by turning slightly.

#### 5.4.4 Cleaning the mixing chamber

Clean the mixing chamber consisting of the chamber tube and the chamber head. Proceed as follows:

- 1. Remove the sealing rings from the chamber head.
- 2. Use diluted mineral acid (HNO<sub>3</sub>, HCl, H<sub>2</sub>SO<sub>4</sub>) or, depending on the analyzed substances an appropriate organic solvent, for cleaning the component.
- 3. If the mixing chamber is cleaned with a diluted mineral acid, wash thoroughly with distilled water after cleaning.

#### 5.4.5 Cleaning the siphon

- 1. Use nitric acid, diluted mineral acid, or an appropriate organic solvent matching the analyzed substances for cleaning the component. Clean the channels and the float tank with a circular brush.
- 2. If the siphon is cleaned with a diluted mineral acid, wash thoroughly with distilled water after cleaning.

#### 5.4.6 Assembling the burner-nebulizer system



#### WARNING

Risk of explosion due to leaking gas connections!

When connecting the supply tubes, ensure correct connection. Insert the sealing rings and test their tightness. Only hand-tighten all screwed connections.



#### CAUTION

Never use the acetylene nitrous oxide flame for sensitivity fine adjustment of the nebulizer! Sudden changes in flow rate may cause a flame flashback into the mixing chamber.

- 1. Check all sealing rings of the chamber head, connections and the nebulizer, replace worn out sealing rings, pull on seals and ensure correct positioning.
- 2. Hold the impeller at its handle (item 3 in Fig. 61 on page 98) and insert it into the mixing chamber tube. Lock by pressing slightly.
- 3. Mate the mixing chamber parts (chamber tube and chamber head), align the sides so that they are flush and screw them together (item 2 in Fig. 61). Ensure that the sealing rings are seated correctly.
- 4. Screw the siphon sensor (item 11 in Fig. 61) into the siphon. Attach the siphon to the chamber head ensuring that the float tank is inclined towards the front (refer to Fig. 60 on page 98 for the correct positioning). Attach the siphon using the clamp screw (item 9 in Fig. 61).
- 5. Attach the safety plug (item 1 in Fig. 61) on the chamber tube.

- 6. Screw the connections for fuel gas and auxiliary oxidant (item 5 in Fig. 61) with the sealing rings into the mixing chamber head. Only tighten the knurled-head screws finger tight.
- 7. Attach the nebulizer (item 8 in Fig. 61) to the chamber head and fasten it using the locking ring (item 6 in Fig. 61).

**Note:** If the nebulizer cannot be stuck easily into the chamber head, slightly grease the sealing rings with the lubricant supplied (Apiezon grease).

- 8. Attach the mixing chamber nebulizer system to the height adjustment using the retaining clip (item 12 in Fig. 60). The white marking must be above the edge of the holding fixture. The plate of the mixing chamber tube must rest evenly on the height adjustment. Screw the knurled head screw at the holding bow tightly.
- 9. Plug the cable of the siphon sensor (item 8 in Fig. 60) into the connection on the sample chamber wall (mind the lug) and screw tight.
- 10. Attach the outlet tube to the outlet connector of the siphon (item 7 in Fig. 60). Route the outlet tube to the waste bottle in a constant downward slope.
- 11. Fill the siphon with water via the mixing chamber tube until the water starts running off via the outlet tube.
- 12. Set the burner on the mixing chamber tube and turn against the 0° stop. Clamp with retaining screw (item 2 in Fig. 60).
- 13. Connect the gas supply:
  - Screw the fuel gas hose (red) onto the connector at the top of the mixing chamber head (item 11 in Fig. 60).
  - Screw the oxidant hose (blue) onto the nebulizer connector (item 10 in Fig. 60).
  - Screw the auxiliary oxidant hose (black) onto the connector on the side of the mixing chamber (item 10 in Fig. 60).

Only tighten the knurled-head screws finger tight.

14. Hang the safety glass in and slide it in front of the burner.

Sensitivity control / adjustment

- 1. In the ASpect LS software for flame technique, use the 🔌 button to open the FLAME / CONTROL window.
- 2. Set the ratio between gas  $C_2H_2$  air in the group field SETTINGS.

#### \land Caution

Sensitivity fine adjustment must not be carried out with  $C_2H_2$  -  $N_2O$  flame. Sudden changes in flow rate may cause a flame flashback into the mixing chamber.

- 3. Align the burner to the height of and parallel to the optical axis.
- 4. Use the [IGNITE FLAME] button to ignite the flame.
- 5. Open the MANUAL OPTIMIZATION tab.
- 6. Select an element line, e.g. Cu324, and click [SET].
- 7. Aspirate a test solution, e.g., Cu / 2 mg/L, via the nebulizer and initiate the continuous display of the measured values by clicking [START]. Evaluate the signal.
- 8. If the sensitivity is too low, adjust the nebulizer in such a way that the absorbance attains its maximum at the selected item line:

Loosen the lock nut (item 2 in Fig. 64).

2 3

- Use the adjustment nut to adjust the depth of the canula (item 3 in Fig. 64).
- 9. After completing the adjustment, secure the adjustment with lock nut (item 2 in Fig. 64).
  - ✓ The burner-nebulizer system is cleaned and installed.
    - 1 Baffle ball
    - 2 Lock nut
    - 3 Adjustment nut for cannula
    - 4 Inner cannula
    - 5 Connection for oxidant

Fig. 64 Components of the nebulizer

#### 5.4.7 Aligning the atomizer within the beam path

The novAA 800 D has an adjustment screw to align the burner-nebulizer system in the sample chamber depth. For adjusting the system, the stop of the tilting bracket is reset.



Fig. 65 Adjustment screw for aligning the atomizer

- 1. In the ASpect LS software for flame technique, use the 🌢 button to open the FLAME / CONTROL window.
- 2. Set the ratio between gas  $C_2H_2$  oxidant (air or  $N_2O$ ) in the group field SETTINGS.
- 3. Use the [IGNITE FLAME] button to ignite the flame.
- 4. Open the MANUAL OPTIMIZATION tab.

- 5. Select an element line, e.g. Cu324, and click [SET].
- 6. Aspirate a test solution, e.g., Cu / 2 mg/L, via the nebulizer and initiate the continuous display of the measured values by clicking [START]. Evaluate the signal.
- 7. Change the setting of the adjustment screw with an Allen key (M 3.0) until the absorbance has reached its maximum for the selected element line.

For the models novAA 800 G + F it is possible to use the factory-set configuration for all measurement tasks.

In addition to that, the control and analysis software ASpect LS automatically sets the height of the atomizer for all models of the novAA 800 product family after selecting the atomization technique in the QUICKSTART window.

#### 5.4.8 Cleaning the sensor for burner detection

A sensor system monitors whether the burner is placed on the mixing chamber neck before igniting the flame. The openings of the sensor system must be cleaned, if

- there are deposits in the opening (e.g. salt incrustation)
- the program produces an error message even though the burner is mounted on the tube of the mixing chamber.
- 1. Hold the burner-nebulizer system tightly, loosen the knurled head screw on the retaining clip of the mixing chamber tube (item 12 in Fig. 60), rotate the retaining clip backwards and remove the system.
- 2. Use a small brush (toothbrush) with alcohol, e.g. isopropyl alcohol, to gently clean the sensor openings.
- 3. Allow the sensor openings to dry.
- 4. Remount the burner-nebulizer system into the height adjustment.



Fig. 66 Openings of the sensor system for the burner detection

## 5.5 Autosampler graphite AS-GF

The following maintenance work must be carried out on the AS-GF:

- Remove any contamination from the sample tray and the casing with a dry cloth on a daily basis
- Clean, shorten, replace the dosing tube
- Replace the dosing syringe
- Clean the housing after the purge cup has overflowed

#### 5.5.1 Purging the dosing tube

The dosing tube must be purged prior to and after work. To do this, purge solution is taken from the storage bottle, pumped via the dosing syringe into the dosing tube and dispensed into the purge cup.

- 1. Switch on the novAA 800 and start the ASpect LS software / graphite furnace technique.
- 2. In ASpect LS use 🔤 to open the AUTOSAMPLER window.
- 3. Use the [WASH] button to start the wash cycle.
- 4. The dosing tube must be dipped into the purge cup until just below the tube guide to ensure that it is sufficiently rinsed during the washing process.

If the dosing tube is not immersed deep enough into the purge cup during the washing process, the autosampler must be realigned in the washing position:

- Click the [ADJUST SAMPLER] button in the FUNCTION TESTS tab.
- Enable the WASH POSITION option in the ALIGNMENT POSITION group field of the ADJUST SAMPLER window. Enter the immersion depth (approx. 40 mm) in the list field of the ALIGNMENT WASH POSITION group field.
- Correct the alignment of the swivel arm with the arrow keys.
- Use the respective buttons to save the settings and close the window.

**Note:** When the ADJUST SAMPLER window is opened the next time, the field DEPTH will show 13 MM instead of the value that was actually saved.

5. The wash cycle can be repeated several times if required.

**Note:** The wash cycle can be defined in the method and thus performed automatically prior to and after the measurement.

If one method is active, the number of wash cycles specified for this method will be executed after pressing the [WASH] button in the AUTOSAMPLER window.

🖶 Autosampler		- 🗆 X
Parameters Techn. parameters Function	tests Positions	
Tracker/Rotator ○ Cup no ● Wash position ○ Tube position ○ Tube position Pipetter Speed: 3 Volume [µL]: 0 Take up Dispense Valve to bottle Reset	Dipping arm Depth [mm]: 0 = Test programs © Test program 1 Test program 2 O Test program 3 Start	Error test Version: 14.599 Tracker/Rotator Tray ident Pipetter (drive) Pipetter (volume) Test Adjust sampler
Wash Initialize		OK Cancel

Fig. 67 AUTOSAMPLER window, FUNCTION TESTS tab



Fig. 68 ADJUST SAMPLER window

#### 5.5.2 Servicing the dosing tube

A defective, kinked or contaminated dosing tube can cause distorted measurement results. Maintenance work is:

- Cleaning the dosing tube
- Shorten the dosing tube
- Replace the dosing tube



Dosing tube on the AS-GF

- 1 Tube holder
- 2 Dosing tube
- 3 Tube holder
- 4 Screw top at the dosing unit
- 5 Retaining screw for the tube guide
- 6 Clamp nut for the tube guide

Fig. 69 Dosing t

Cleaning the dosing tube

The dosing tube requires cleaning, dependent on the sample material, when:

- The pH levels of the sample, the wash liquid and the air bubble are blurred, or if the bubble is segmented.
- The sample is carried over because the tube's interior is contaminated.

An 8 to 13% sodium hypochlorite solution (NaOCI) is recommended as a cleaning solution. Repeat the cleaning process several times, if necessary.

	1. Fill the sodium hypochlorite solution into a 5 mL special cup and mount tray position 101 with it.
	2. Switch on the novAA 800 and start the ASpect LS software.
	3. In ASpect LS use to open the AUTOSAMPLER window. Open the FUNCTION TESTS tab (Fig. 67 on page 105).
	4. Enter "101" in the TRACKER/ROTATOR field and enable the option CUP NO. The autosampler arm moves to position "101".
	5. Use the arrow keys to lower the autosampler arm into the special cup (approx. 50 mm) in the DEPTH list field of the DIPPING ARM area.
	<b>Note:</b> The autosampler is only lowered if the arrow keys are used. After entering the value directly into the list field, click the arrow keys once again!
	6. Use the arrow keys to set the volume to be taken (100 to 200 $\mu$ L) in the VOLUME [ $\mu$ L] list field of the PIPETTER area. The volume can be set in steps of 50 $\mu$ L.
	7. Press the button [TAKE UP]. The autosampler fills the dosing tube with the cleaning liquid.
	8. Allow the cleaning liquid to react for approx. 20 min.
	9. In the area TRACKER/ROTATOR enable the option WASH POSITION.
	10. The autosampler arm moves to the wash position.
	11. Use the arrow keys to lower the autosampler arm into the purge cup (ap- prox. 40 mm) in the DEPTH list field of the DIPPING ARM area. When entering the value directly into the list field, click the arrow keys once again!
	12. Click the [DISPENSE] button to empty the dosing tube into the purge cup.
	13. Start 5 wash cycles. (Click the [WASH] button 5 times).
	✓ The dosing tube is cleaned.
Shortening the dosing tube	1. Loosen the clamp nut at the tube guide (item 6 in Fig. 69) and remove the dosing tube by pulling upwards.
	2. Cut approx. 70 mm of the dosing tube with a razor blade or a scalpel at an angle of 10° to 15°.
	3. Push the dosing tube as far as possible into the tube guide until the dosing tube protrudes by approx. 8 mm beyond the bottom.
	4. Lock the dosing tube with the clamp nut.
	5. Readjust the injection depth of the sample ( $\rightarrow$ section "Adjusting the sampler" on page 70).
	✓ The autosampler is ready for operation again after contaminated or damaged parts of the tube were removed.
Replace the dosing tube	1. Loosen the clamp nut at the tube guide (item 6 in Fig. 69) and pull out the tube. Remove the tube from the tube holders at the autosampler arm and the back of the autosampler (items 1 and 3 in Fig. 69).
	2. Detach the screw top from the T valve of the dosing unit (item 4 in Fig. 69 ).
	3. Screw the new dosing tube to the valve and feed it through the tube holders.

- 4. Push the dosing tube as far as possible into the tube guide until the dosing tube protrudes by 8 mm underneath. Lock with the clamp nut.
- 5. Readjust the injection depth of the sample ( $\rightarrow$  section "Adjusting the sampler" on page 70).
  - ✓ The autosampler is ready for operation with a new dosing tube.

#### 5.5.3 Replacing the dosing syringe

The details below apply to the samplers AS-GF (graphite furnace technique) and AS-FD (flame technique). The dosing units only differ in the size of the dosing syringe (500 or 5000  $\mu$ L).



- 1 T valve
- 2 Dosing syringe, consisting of piston and glass cylinder
- 3 Attachment screw
- 4 Drive rod

Fig. 70 Dosing unit at AS-GF and AS-FD

- 1. Switch on the novAA 800 and start the ASpect LS software. Select the technique in the QUICKSTART window: GRAPHITE FURNACE (AS-GF) or FLAME (AS-FD).
- 2. Use 🛱 to open the AUTOSAMPLER window. Open the FUNCTION TESTS tab.
- 3. Use the arrow keys to set a volume to be taken in the VOLUME [ $\mu$ L] list field in the PIPETTER area (AS-GF: 500  $\mu$ L; AS-FD: 5000  $\mu$ L). Increase the speed to 6-7.
- 4. Press the button [TAKE UP]. The piston of the dosing syringe moves down.
- 5. Unscrew the fixing screw (item 3 in Fig. 70).
- 6. Unscrew and remove the dosing syringe (item 2 in Fig. 70).
- 7. Screw the new dosing syringe to the valve.
- 8. Carefully pull the piston down until the eyelet at the piston end is aligned with the hole in the drive rod.

Screw the piston with the attachment screw finger-tight to the drive rod.

#### **i** Caution

Excessive force can lead to material damage! Do not tighten the screw too much.

- 9. Click the [INITIALIZE] button in the AUTOSAMPLER window. The piston of the dosing unit returns to the initial position.
  - ✓ The autosampler is ready for operation with a new dosing syringe.
### 5.5.4 Cleaning the autosampler after cup overflow

If during the process a purge cup has overflowed, immediately interrupt the process and clean the device.

- 1. Instantly abort the analysis process.
- 2. Take up the liquid with cellulose wadding or cloth. Wipe the device surface dry.
- 3. Ensure that the outlet can be drained, i.e., remove any kinks in the outlet tube and make sure that the outlet tube does not dip into the liquid in the waste bottle.
  - ✓ The analysis process can be continued.

# 5.6 Autosamplers AS-F, AS-FD

Contamination on the tray and the casing can be removed with a dry cloth on a daily basis as required. In addition, according to conditions:

- Wash the sample path
- Wash the mixing cup
- Replace the cannula(s) at the autosampler arm
- Replace the aspiration tube and dosing tube
- Replace the dosing syringe (→ section "Replacing the dosing syringe" on page 108)
- Clean, after a wash or mixing cup has overflowed.

#### 5.6.1 Washing the sample path

- 1. Use the 🔮 button to open the FLAME window in the software Aspect LS and ignite the flame.
- 2. Use the 🖴 button to open the AUTOSAMPLER window.
- 3. On the PARAMETER tab, set approx. 60 s in the WASH TIME input field.
- 4. Use the [WASH] button to start the wash cycle.

The cannula of the autosampler dips into the purge cup. The wash liquid is aspirated through the system.

### 5.6.2 Washing the mixing cup of the AS-FD

The mixing cup must be washed before and after the operation to prevent adhesion and scaling.

Before preparing the first standard / first sample the mixing cup is washed automatically. Further washing processes might be useful during continuous operation.

Washing the mixing cup prior to and after the measurement

- 1. In ASpect LS use 🔤 to open the AUTOSAMPLER window.
- 2. On the PARAMETER tab in the WASH MIX CUP group, enter a volume of 25 mL.
- 3. Use the [START] button to start the wash cycle.
- 4. The wash cycle can be repeated several times if required.

25 mL of washing liquid is dispensed from the storage bottle into the mixing cup and automatically drained off afterwards.

Washing the system before shutting down the device for an extended period If salts were added to the diluent (bidistilled or acidic bidistilled water), the dosing unit and valve must be washed with methanol or ethanol before the device is put out of service for an extended period of time. Otherwise scaling and blocking may also occur.

- 1. Fill the storage bottle for the diluent with methanol or ethanol.
- 2. Perform the wash cycle as described in Section "Washing the system prior to and after the measurement". Repeat the washing process several times.

### 5.6.3 Replacing the cannulas and guide on the autosampler arm of the AS-FD

The cannulas and guide must be replaced in case of significant contamination or mechanical damage (detectable by large standard deviations in the measurements).

- 1. Pull off the hoses from the cannulas.
- 2. Loosen the fixing screw on the autosampler arm.
- 3. Pull the cannula guide with cannulas upwards and out.
- 4. Fit the guide with the new cannulas into the autosampler arm and fix in place with the locking screw.

#### Caution! Risk of fracture!

Set the cannula height so that the cannulas terminate 1-2 mm above the block with the wash and mixing cup.

5. Plug the sample intake tube onto the thinner cannula. Plug the dosing tube for the diluent onto the thicker cannula.

### 5.6.4 Replacing the cannula on the autosampler arm of the AS-F

The cannula for picking up the sample (thin cannula) must be replaced in case of significant contamination or mechanical damage (detectable by large standard deviations in the measurements).

- 1. Pull the intake tube off the cannula.
- 2. Loosen the lock screw at the autosampler arm and pull out the guide with the cannula.
- 3. Insert the new guide with the cannula and fix with the clamp nut.

#### Caution! Risk of fracture!

Set the cannula height so that the cannula terminates 1 - 2 mm above the block with the wash and mixing cup.

4. Plug the intake tube onto the new cannula.

### 5.6.5 Replacing the intake tube

If the intake tube is contaminated, it must be replaced.

- 1. Remove the aspiration tube from the autosampler arm cannula and then from the nebulizer cannula.
- 2. The aspiration tube is equipped with a silicon adapter tube on both ends. Insert the longer tube adapter on the cannula and the shorter tube adapter on the nebulizer.

#### Caution!

Do not mix up the two connections. This may cause leaks in the system.

#### 5.6.6 Replacing the tube set on the AS-FD

- 1. Pull the dosing tube for the diluent off the thicker cannula on the autosampler arm and feed it through the tube guide (item 6 in Fig. 52 on page 78).
- 2. Detach the tube for the washing liquid at the rear of the autosampler (item 5 in Fig. 52 on page 78).
- 3. Pull the encased tubes out of the attachment lug at the rear of the autosampler.
- 4. Pull the tube for the washing liquid off the storage bottle.
- 5. Unscrew the dosing tube from the change-over valve (item 3 in Fig. 53 on page 79).
- 6. Screw the new tube set with dosing tube (marking "1") to the change-over valve and attach the encased tubes with the attachment lug to the rear of the autosampler.
- 7. Insert the tube with the marking "2" into the storage bottle for the washing liquid.
- 8. Screw the tube for the washing liquid to the rear of the autosampler.
- 9. Slide the other end of the dosing tube through the tube guide onto the thicker cannula of the autosampler arm.

### 5.6.7 Cleaning up after cup overflow

If the purge cup or mixing cup (when using the AS-FD) overflows during the analysis process, immediately interrupt the analysis and clean the device.

- 1. Stop the measuring process immediately.
- 2. Take up the liquid with cellulose wadding or cloth. Wipe the surface dry.
- 3. Use to open the AUTOSAMPLER window. Open the FUNCTION TESTS tab. Check the MIX CUP PUMP checkbox in the PUMPS section to start the pump. Run the pump until the liquid is pumped out. If necessary, activate and deactivate the pump several times.

# 5.7 Cooling unit KM 5

(Selected technique: graphite furnace technique)

**Note:** Please observe the maintenance and care instructions in the separate operating instructions for the cooling unit.

Maintenance work	•	Check the level and cleanliness of the cooling liquid every three months.
	•	If air bubbles occur in the cooling circuit (noticeable by the sound) check the water level.
Empty offline	1.	Keep a receptacle with a capacity of 5 L readily available.
	2.	When the novAA 800 is switched off position the return flow tube of the KM 5 (connection is indicated by on the KM 5) in the receptacle.
	3.	Switch on the KM 5.
		✓ The water cooler is pumped out (emptied).
Filling and venting	1.	Open the top of the KM 5 and remove the lid of the fill opening.
	2.	Fill up with 5 liters of softened water using a funnel (up to approx. 5 cm below the lid).
	3.	Place the back flow tube in the coolant container of the KM 5.
	4.	Switch on the KM 5. Allow the water cooler pump to run until the returning water is free of air. Switch the KM 5 on and off several times, as required.
	5.	Switch off the KM 5. Reconnect the back flow tube to the KM 5. Close the filling opening and the lid of the KM 5.

# 5.8 Piston compressor PLANET L-S50-15

(Selected technique: flame technique)

**Note:** Please observe the maintenance and care instructions in the separate operating instructions for the compressor.

• Pressure cylinder and fluid separator on the pressure reducer:

Drain oily condensate from the pressure cylinder (furnace) every week by opening the drain cock.

#### ▲ Caution! Risk of splashing!

The furnace is under pressure. To avoid splashing of liquid, attach a hose to the cock and slowly open the cock to carefully drain the liquid into a waste bottle.

Drain oily condensation water from the pressure reducer every week by pressing the pin at the bottom of the liquid separator.

Suction filter:

Check the filter every month. Clean or replace the filter every six month.

• Oil:

Only use the special oil SE-32! Dispose of the used oil according to the applicable regulations.

Check the oil level on the inspection glass every week. Refill oil, if necessary. Change the oil every 12 months.

- Unscrew the 4 screws and remove the ribbed cover to do this.
- Tilt the tank far enough to allow the oil to drain from the tank. Hold the motor block with one hand while draining the oil to prevent it from falling out.
- Remove any contaminations from the housing.
- Check the O ring on the ribbed cover and replace it, if required. Clean the sealing surfaces.
- Top up about 0.6 L of oil (SE-32).
- Remount the ribbed cover. Check the tightness of the ribbed cover when the device is in operation.

# 6 Troubleshooting

# 6.1 Troubleshooting according to software messages

The following chapter describes a number of possible problems that the user can partially remedy independently. If such problems occur frequently, the operator must inform the Analytik Jena service department.

A system monitoring process is initiated as soon as the novAA 800 is switched on. Any errors that occur are displayed in a window after start-up.

The user must acknowledge the error messages by clicking the [OK] button.



### NOTICE

Risk of device damage!

If the errors below cannot be remedied using the corresponding troubleshooting notes, the operator must inform the service department at Analytik Jena. This also applies for the repeated occurrence of individual faults.

Error code	Error message	
4111	Flame does not ignite – fuel/oxidant pressure may be too low or flame sensor detects light; Eliminate problem and retry!	
Cause		Remedy
<ul> <li>Error o</li> </ul>	n the gas supply	<ul> <li>Check the gas supply (air, fuel gas)</li> </ul>
4231 4234	No argon pressure (status) No aux. gas pressure (status)	
Cause		Remedy
<ul> <li>Gas sup device</li> </ul>	oply closed before reaching the connection	<ul> <li>Check the gas supply, open the gas sup- ply before the gas connection</li> </ul>
4232	Toroidal transformer temperatur	e error (status)!
Cause		Remedy
<ul> <li>Transfe</li> </ul>	ormer temperature too high	<ul> <li>Allow the device to cool down for at least 1 h. If necessary, reduce the thermal load in the temperature-time program</li> </ul>
4233	Cooling system sensor error (stat	us)
Cause		Remedy
<ul> <li>Insufficer</li> <li>reserve</li> </ul>	ient filling level of the coolant ir.	<ul> <li>Check the water level in the cooling unit and top up the cooling water, if required.</li> </ul>

Error code	Error message	
4301	Firmware update communicatio	ns error
Cause		Remedy
<ul> <li>Firmwa</li> </ul>	are update has failed	<ul> <li>Repeat the firmware update</li> </ul>
		<ul> <li>Inform service</li> </ul>
5003	Line source signal or emission si	gnal too small
Cause		Remedy
<ul> <li>HCL to</li> </ul>	o weak	<ul> <li>Check the calibration state of the HCL and the atomizer</li> </ul>
		<ul> <li>Increase the slit width and the lamp current</li> </ul>
		<ul> <li>Choose a more energy intensive HCL line</li> </ul>
5005	Energy drift or energy fluctuatio	ns too big
Cause		Remedy
<ul> <li>Fluctua big</li> </ul>	ations of the HCL energy are too	<ul> <li>Start up the HCL and wait for 5 to 15 minutes before manually repeating an energy alignment and a peak search</li> <li>Check the calibration state of the HCL</li> </ul>
		<ul> <li>Increase the slit width and the lamp current</li> </ul>
		• Choose a more energy intensive HCL line
		<ul> <li>Check the aging (blackening) of the HCL and replace the HCL, if required</li> </ul>
5006	Background radiator signal too l	ow
Cause		Remedy
Energy	of the $D_2$ HCL too low	<ul> <li>Increase the slit width or select a HCL analysis line with a shorter wavelength</li> <li>Check the alignment and aging (blackening) of the D<sub>2</sub>-HCL</li> </ul>
		<ul> <li>Check the calibration distance of the atomizer</li> </ul>
		<ul> <li>If necessary, do without the background compensation</li> </ul>

Error code	Error message	
5008 Energy drift or energy fluctuatio		ns in the background radiator too big
Cause		Remedy
<ul> <li>Fluctua too big</li> </ul>	ations of the $D_2$ -HCL energy are	<ul> <li>Increase the slit width or select an analysis line with a shorter wavelength</li> <li>Check the alignment and aging (blackening) of the D<sub>2</sub>-HCL</li> <li>Check the calibration distance of the atomizer</li> </ul>
5009	Too much energy during the zero	balance (AZ phase)
Cause		Remedy
<ul> <li>Drift of</li> <li>Inapprend the Inapprend the Inapprend</li></ul>	f the HCL energy level too big opriate moment in the measure- process for the AZ phase	<ul> <li>Start up the HCL and wait for 5 to 15 minutes before manually repeating an energy alignment and a peak search</li> </ul>
5010	Too little energy during the zero	balance (AZ phase)
Cause		Remedy
<ul> <li>Drift of</li> <li>AZ pha spec. A</li> <li>Atomiz beam p</li> </ul>	f the HCL energy level too big ise disturbed (fogged window, un- IBS signal) zer is not ideally aligned with the path	<ul> <li>Start up the HCL and wait for 5 to 15 minutes before manually repeating an energy alignment and a peak search</li> <li>Cleaning the windows</li> <li>Check the calibration distance of the atomizer</li> </ul>
5012	Baseline drift during the AZ phas	e (total absorption)
Cause		Remedy
<ul> <li>Drift of</li> </ul>	f the HCL energy level too big	<ul> <li>Start up the HCL and wait for 5 to 15 minutes before manually repeating an energy alignment and a peak search</li> </ul>
5215	Cooling water flow too low!	
Cause		Remedy
<ul> <li>The flo</li> <li>Partial channe</li> </ul>	w of the cooling water is too low obstruction of the cooling water els	<ul> <li>Check the cooling water level in the cooling unit</li> <li>Top up the cooling water</li> <li>Contact the customer service in case of obstructions</li> </ul>

# 6.2 Equipment faults and analytical problems

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

No signal or sensitivity is too low	
Cause	Remedy
<ul> <li>Atomization unit is inappropriately aligned in the beam path</li> </ul>	<ul> <li>Check the height adjustment</li> <li>On the novAA 800 D, correct the depth adjustment of the burner-nebulizer system using the adjustment screw</li> </ul>
<ul> <li>Leak or obstruction in the sample introduction system</li> </ul>	<ul> <li>Check the cannula and dosing tube for deposits, kinks and cracks, and clean or replace them, if required</li> </ul>
<ul> <li>Sample is not injected correctly into the graphite tube (graphite furnace technique)</li> </ul>	<ul> <li>Check the pipetting, adjust the autosampler</li> </ul>
<ul> <li>Nebulizer clogged (flame technique)</li> </ul>	<ul> <li>Check the flow through the nebulizer and clean it</li> <li>If necessary, filter the sample solutions</li> </ul>
<ul> <li>Nebulizer gas is set too low (flame technique)</li> </ul>	<ul> <li>Optimize the nebulizer flow (air / N<sub>2</sub>O)</li> </ul>
The measured value is too low	
Cause	Remedy
<ul> <li>Calibration is incorrect</li> </ul>	<ul> <li>Check the calibration solutions</li> </ul>
<ul> <li>Substances with low solubility lead to poorer results</li> <li>Substances with low solubility are not completely digested</li> </ul>	<ul> <li>Optimize sample preparation</li> </ul>
<ul> <li>Formation of sparingly soluble compounds in the flame (oxides, carbides, phosphates)</li> </ul>	<ul> <li>Increase the flame temperature, e.g. by changing over to the nitrous-oxide-acetylene flame</li> <li>Addition of "releasing agents" such as lanthanum chloride that absorb the disturbing phosphates, for example</li> </ul>
<ul> <li>Volatile substances escape during sample preparation</li> </ul>	<ul> <li>Optimize sample preparation</li> </ul>
<ul> <li>Contamination / carry-over in the cal-zero solution</li> </ul>	<ul> <li>Remedy the cause for carry-over / contamination</li> </ul>
<ul> <li>The sample solution is viscous / has a higher density / other sur- face tension than the calibration solution</li> </ul>	<ul> <li>1st Adjust the matrix (add matrix to the calibration solutions or dilute it)</li> <li>2nd Standard addition</li> </ul>

•	Analytes evaporate too early / too late (graphite furnace technique)	•	Perform a standard addition Optimize the furnace program (e.g. by reducing the pyrolysis temperature)
•	The analyte is an alkali metal (or an easily excitable atomic line)	•	Alkali effect, addition of ionization buffers that are ionized instead of the analyte
•	Slight offset of the peak position		Perform a wavelength correction

The measured value is too high	
Cause	Remedy
<ul> <li>Calibration is incorrect</li> </ul>	<ul> <li>Check the calibration solutions</li> </ul>
Contamination / carry-over	<ul> <li>Find causes and correct them</li> </ul>
<ul> <li>Warm-up phase of the device was not observed</li> </ul>	<ul> <li>Allow the flame to stabilize for a longer period after igniting it before performing the calibration</li> </ul>
<ul> <li>The sample foams when shaken</li> </ul>	<ul> <li>There are surface-active substances in the measurement solutions</li> <li>1. Optimize sample preparation</li> <li>2. Add the surface-active substances to the calibration solutions, too</li> </ul>
<ul> <li>Line superposition with matrix element</li> </ul>	<ul> <li>Use matrix modifiers in the graphite furnace technique, optimize the furnace program (thermal pretreatment)</li> <li>Optimization of the flame temperature</li> </ul>
<ul> <li>The sample solution is viscous / has a higher density / other sur- face tension than the calibration solution</li> </ul>	<ul> <li>1st Adjust the matrix (add matrix to the calibration solutions or dilute it)</li> <li>2nd Standard addition</li> </ul>
Precision is poor	
Cause	Remedy
<ul> <li>Dispersion of solid matrix compo- nents (soot, oxide, salt particles) and gases (solvent vapor)</li> </ul>	<ul> <li>Graphite furnace technique:</li> <li>Optimize the furnace program (drying phase, thermal pretreatment)</li> <li>Use a matrix modifier</li> <li>Flame technique:</li> <li>In case of sooting: Increase the flame temperature (more air), use the acetylene-nitrous-oxide flame</li> </ul>
<ul> <li>Contamination / carry-over in the graphite tube (graphite furnace technique)</li> </ul>	<ul><li>Bake out the graphite tube to clean it</li><li>Optimize the furnace program (cleaning phase)</li></ul>
<ul> <li>Purging time between two sam- ples is too short (flame technique)</li> </ul>	<ul> <li>Extend the purging time</li> </ul>
<ul> <li>Fluctuations in the burner temper- ature (flame technique)</li> </ul>	<ul> <li>Use the injection module SFS 6</li> </ul>

<ul> <li>Contamination / carry-over in the nebulizer (flame technique)</li> </ul>	<ul> <li>Check the flow through the nebulizer and clean it</li> </ul>
	<ul> <li>If necessary, filter the sample solutions</li> </ul>
<ul> <li>The nebulizer gas flow is not opti- mal (flame technique)</li> </ul>	<ul> <li>Optimize the nebulizer gas flow</li> </ul>
Drift	
Cause	Remedy
<ul> <li>Atmospheric oxygen in the graph- ite tube at the beginning of the measurement</li> </ul>	<ul> <li>Perform a formation of the graphite tube be- fore starting the measurement</li> </ul>

# 7 Transport and storage

# 7.1 Preparing the novAA 800 for transport

#### Tools

- 4 handles (supplied with the device)
- 17-mm open-end wrench



### CAUTION

Risk of injury!

The various different models of the novAA 800 product family weigh between 95 kg and 130 kg. The device must be transported by at least 4 persons using the fixed screw-in carrying handles.



# CAUTION

Risk of burns from touching hot surfaces! Observe the required cooling times of the novAA 800 before preparing the device for transport.



### NOTICE

Using improper packaging material and omitting the transport locks may cause damage to the device!

Always use the original packaging when transporting the novAA 800. When transporting the novAA 800 D and G, insert the transport lock into the sample chamber to secure the graphite furnace in parking position. In addition to that, use a transport lock to secure the monochromator.

- Close the control and analysis software ASpect LS. Observe the sequence for switching off the PC and the novAA 800 and turn them off (→ section "Switching off sequence" on page 83).
- 2. Detach all components and accessories ( $\rightarrow$  section "Installation and initial start-up" on page 38). Remove the autosampler from the sample chamber.
- 3. Flame technique: Remove the outlet tube of the siphon and the safety glass pane.
- 4. Empty the waste bottle and dispose of the waste.
- 5. Close the gas supply upstream of the device connections.
- 6. Detach the gas connectors on the rear of the novAA 800:
  - Manually detach the gas connectors for the inert gas (argon) and the auxiliary gas, if applicable.
  - Manually detach the tubes for compressed air and nitrous oxide.
  - For the acetylene gas connector, use a 17-mm open-end wrench. Left hand thread!
- 7. Undo the electrical connections.
- 8. Graphite furnace technique: Detach the quick-release connectors of the coolant hoses on the novAA 800. Drain the mobile cooling unit ( $\rightarrow$  section "Cooling unit KM 5" on page 112).

- 9. Insert the transport lock for the monochromator ( $\rightarrow$  section "Removing the transport locks" on page 51):
  - Remove the device cover.
  - Screw the transport lock that is marked red into the mesh lever.
  - Reattach the device cover.
- 10. novAA 800 D + G: Use the pivoting mechanism to tilt the graphite furnace backwards. Insert the transport lock into the opening in the back of the sample chamber so that the wedge locks the graphite furnace in parking position.



Fig. 71 Inserting the transport locks for the graphite furnace

- 11. Remove the four plugs (provided with the device) from the holes for the handles on both sides of the device and keep retain them for future use.
- 12. Screw the four handles securely into the holes as far as the end stops.
  - ✓ The novAA 800 is prepared for transport

# 7.2 Ambient conditions for transport and storage

Observe the safety instructions in the section "Safety instructions, transport and installation" on page 11. Transport the novAA 800 and its components with care to prevent damage from impact or vibration. The device should be transported in such a way that major temperature fluctuations are avoided and the formation of condensate is thus prevented. The following ambient conditions must be met when transporting and storing the device:

Temperature range	
Transport	-40 °C to +70 °C
Storage	+5 °C to +40 °C
Max. humidity:	90% at 40 ℃

If the novAA 800 and add-on devices are not installed immediately after delivery or are not required for a prolonged period of time, store them in their original packaging. A suitable desiccant should be added to the packaging to prevent damage from moisture.

# 8 Disposal

Atom absorption spectrometry usually creates only liquid waste. The liquid waste contains metal ions or heavy metal ions, but mostly different mineral acids which were used during 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.

At the end of its service life, the novAA 800 and all its electronic components must be disposed of as electronic waste in accordance with the applicable regulations.

Dispose of the hollow cathode lamps in accordance with the local requirements or contact the Analytik Jena service department.

# 9 Specification

# 9.1 Technical data

# 9.1.1 Data on the novAA 800

Techniques	<ul> <li>Graphite furnace tech correction</li> </ul>	nnique in single-beam operation with deuterium background		
	<ul> <li>Flame technique in s correction</li> </ul>	ingle or double-beam operation with deuterium background		
	<ul> <li>Hydride and mercury um background corre</li> </ul>	cold vapor technique in single-beam operation with deuteri-		
	<ul> <li>HydrEA technique in</li> </ul>	single-beam operation with deuterium background correction		
Background correction	<ul> <li>Deuterium backgroui</li> </ul>	nd correction with D <sub>2</sub> -HCL		
Photometer	<ul> <li>Dual-beam arrangement with beam splitter and rotating sector mirror for coupling in the reference beam path</li> </ul>			
	<ul> <li>High light yield and base line stability</li> </ul>			
	<ul> <li>Quartz-improved mirror optics</li> </ul>			
	<ul> <li>Silicon-hydride receiver S 12749</li> </ul>			
	<ul> <li>Optics purging: Optic eration in environme Purge Kit – APK).</li> </ul>	onal purging of the optics with purified compressed air for op- ents with large quantities of dust (in combination with an Air		
Monochromator	Installation	Modified Czerny-Turner layout with an flat hologrid, auto- matic setting of the wavelength and the slit		
	Wavelength range	185 to 900 nm		
	Slit width	0.2 nm, 0.3 nm, 0.5 nm, 0.8 nm, 1.2 nm		
Lamp turret for HCL	PC-controlled 8-lamp turn of coded lamps.	ret for fully automated operation with a write/read unit for the use		
Hollow-cathode lamps	The use of uncoded lamp	s is possible.		
HCL, coded	Lamp type: Glow discharge lamps for 68 elements with line radiation in the UV/VIS range			
	Lamp current	2 to 20 mA		
	Mode	electrical timing 50 Hz		
	Power supply	2 power packs, electrically stabilized		
		- For active lamps		
		- For preheating		

Deuterium hollow- cathode lamp D <sub>2</sub> -HCL	Lamp type: Glow discharge lamp with continuum radiation in the UV range		
	Lamp current	30 mA	
	Mode	electrical timing 50 Hz	
Analytical operating modes in absorption	Total absorption Specific and unspecific absor	ption	
Display types	Absorbance	-0.01 to 3.00	
	Concentration	Value range: 5 characters (0.0001 to 99999), unit freely se- lectable	
	Emission	0 to 1; possible in flame mode	
	Energy	0 to 2,000,000 counts	
	normalized intensity	0 % to 100 %	
Processing of the meas- ured values	Measurement frequency (single value sequence)	Single-beam operation50 HzDual-beam operation25 Hz	
	Signal analy- sis,integration type	Mean value Repeated mean value Maximum value: Maximum value of the absorbance Integral value: time-integrated absorbance	
	Integration time	0.1 to 600 s	
	Autozero (AZ measuring time)	0.1 to 600 s	
	Delay	0 to 600 s	
	Energy measuring time	0.3 s	
	Smoothes	three stages: off ► low ► high	
	Types of displaying the measured values	Absorbance, emission, concentration	
	Number of digits	3, 4 or 5	
	Units of concentration	mg/L, μg/mL, ng/mL, μg/L, ng/L or user defined	
	Results display window	Alphanumerical values Bar chart of integrated values (bar graph) Chronological sequence of the single peaks Overlapping peaks Graphical peak overview	
	Special windows	Temperature-time program (furnace program) Optimization of the furnace program Mercury / hydride report Concentration values in the reference curve Peak plots with variable integration limits	
	QC window (Quality Check)	QC blank – Blank QC chart QC control samples – Mean value chart	

	QC duplicate measurement sample/matrix
	– Differences chart (trend chart)
	– Range chart
	– Precision chart (SD chart)
	QC spike sample
	– Percentage recovery chart
Statistical methods	Sigma statistics
	<ul> <li>Mean value with standard deviation (SD) and relative</li> </ul>
	standard deviation (RSD)
	Median statistics
	– Median value with range (R) and relative range (R %)
Confidence interval	Choose between: absolute, relative or interruptible
	Selectable confidence interval:
	68.3 % (1 <b>σ</b> )
	90 % (1.6 <b>o</b> )
	95.4 %(2 σ)
	99 % (2.6 <b>o</b> )
	99.7 % (3 <b>o</b> )
	99.9 % (3.6 <del>0</del> )
Calibration techniques	Standard calibration (recalibration), Bracketing calibration,
	Standard addition, Addition calibration
Fit reference curve	Linear, variable weighting functions
	Non-linear, variable weighting functions
Number of standards	1 to 30
Number of addition con-	1 to 30
centrations	
Recalibration	Two-point recalibration with display of the recalibration fac-
	tor
Supply voltage	230 V ~
Frequency	50 / 60 Hz
Mains fuse (installation in	35 A, safety fuse, slow blow, single phased
the building)	
Power consumption	2600 VA (basic device: 1400 VA + output socket: 1200 VA)
Maximum current con-	28 A for a period of 8 s or 40 A for 1 s
sumption	
Output socket	Same as input voltage
	For connection of accessories: PC, monitor, printer, hydride
	system, cooling unit
Overvoltage category	II according to DIN EN 61010-1
Degree of contamination	2 according to DIN EN 61010-1
Safety class	
Drotoction type	ID 20
Frotection type	IF ZU

Calibration

#### Power supply novAA 800 D + G

#### Instrument fuses

The fuses of the power supply may only be changed by service engineers from Analytik Jena or by technical personnel authorized by Analytik Jena.

Fittings for gL-fuses (10×38 mm<sup>2</sup>) according to 60947-3.

Fuse number	Туре	Protected circuit
F1	25 A/T	Power supply
F2	25 A/T	Power supply

#### Fittings for G-fuses (5×20 mm<sup>2</sup>) according to IEC 60127.

Fuse number	Туре	Protected circuit
F3	T 6.3 A/H	Socket for external accessories
F4	T 6.3 A/H	Socket for external accessories
F5	T 2.5 A/H	Transformer, primary side, NTL
F6	T 2.5 A/H	Transformer, primary side, NTL
F7	T 0.08 A	D <sub>2</sub> -HCL
F8	T 0.25 A	HCLs
F9	T 3.15 A	Filament

#### Fuse for the furnace

Fuse number	Туре	Protected circuit
F1 internal	TR5-T 100 mA	Measuring lead, graphite tube furnace
Supply voltage	230 V ~	
Frequency	50 / 60 Hz	
Mains fuse	16 A, single phase	
installation in the		
building		
Power consumption	1350 VA (basic de	vice: 150 VA + output socket: 1200 VA)
Output socket	Same as input volt	age
	For connection of a	accessories: PC, monitor, printer, hydride
	system	
Overvoltage category	II according to DIN	EN 61010-1
Degree of contamination	2 according to DIN	EN 61010-1
Safety class	I	
Protection type	IP 20	

Power supply novAA 800 F

Instrument fuses	Fittings for G-fuses ( $5 \times 20 \text{ mm}^2$ ) according to IEC 60127.				
	Fuse number	Туре	Protected circuit		
	F1	T 6.3 A/H	Socket for external accessories		
	F2	T 6.3 A/H	Socket for external accessories		
	F3	T 2.5 A/H	Transformer on the primary side, SNT		
	F4	T 2.5 A/H	Transformer on the primary side, SNT		
	F5	T 0.08 A	D <sub>2</sub> -HCL		
	F6	T 0.25 A	HCLs		
	F8	T 3.15 A	Filament		
Ambient conditions	Compliant with DIN ISO 9002	Compliant with DIN ISO 90022-2:2003 / 01			
	Corrosion protection	The device is corrosion-proof for the samples used in the analysis			
	Working temperature	+5 °C to +40 °C			
	Humidity during operation	Max. 90 % at +40 °C			
	Transport temperature (desiccant)	-40 °C to +70 °C			
	Air pressure	0.7 bar to 1.06 ba	r		
	Recommended max. operating altitude	2000 m			
	The ambient conditions for the novAA 800 are identical for operation and storage.				
Dimensions and weights	The models of the novAA 80 weights.	0 product family hav	e identical dimensions but different		
	Mass	novAA 800 D 130	kg		
		novAA 800 G 125	kg		
	Dimensions	820 mm x 600 mm	«y n x 770 mm		
	(W x H x D):	020 mm x 000 mm			
	Transport of device	Mandatory use of rying handles.	the accompanying, securely screwed on car-		

# 9.1.2 Minimum requirements of the ASpect LS software

Computer	Resolution 1280x1024 pixels or higher
	Mouse / trackball
	2 x USB-2.0 interfaces
Operating system	PC with Windows 7, 8.1 or 10 (32 bit or 64 bit)

# 9.1.3 Data for the graphite tube technique

### Graphite tube furnace

Sample type	Dissolved	
Tube type	IC tube (wall atomization) Graphite tube with PIN platform All types of tube have pyro-coating.	
Sample volume	Max. 40 $\mu$ L (graphite tube with PIN platform) Max. 50 $\mu$ L (IC tube)	
Temperature setting	Temperature can be set between room temperature and $3000 ^\circ$ C, in steps of 0.5 $^\circ$ C	
Temperature-time programming (furnace program)	Up to 20 steps can be freely programmed within determined limits, 0 to 999 s/step, in intervals of 1 s Temperature increase (Ramp): 1 °C/s up to 1200 °C/s linear and No Power (NP) Control of inert gas and auxiliary gas Inserting injection and enrichment steps Determining the starting point for autozero and integration	
Cooling water	Min. 2.5 L/min, sediment-free 20 to 40 °C	
Inert gas	Argon 4.8 and superiorPermitted components:Oxygen $\leq$ 3 ppmnitrogen $\leq$ 10 ppmhydrocarbons $\leq$ 0.5 ppmhumidity $\leq$ 5 ppmConsumption:Max. 2 L/min(depending on the temperature-time program)Inlet pressure:600 to 700 kPa	
	Auxiliary gas: Compressed air, oil-free, grease-free, particle- free Inlet pressure: 600 to 700 kPa	
Safety circuits ensuring protection against	Overheating of the graphite furnace transformer Graphite tube rupture Overheating of the graphite furnace (switch-off at T $\ge$ 100 °C) Operation with an opened graphite furnace Operation with insufficient cooling water levels Operation with insufficient inlet pressure of the inert gas	
Software-controlled height	t adjustment of the graphite furnace in the beam path	
Height	4 to 18 mm, automated	
Depth	Factory preset	

Adjusting the furnace

# 9.1.4 Data for the flame technique

Types of flame	Acetylene-air flame (standard) Acetylene-nitrous-oxide flame for elements that cannot be atomized easily, such as boron, aluminum or silicon				
	Propane/air flame on re	Propane/air flame on request			
	Acetylene/air	One-slit burner 50 mm, coded (standard);;;;;One-slit burner 100 mm, coded (optional)			
	Acetylene/nitrous ox- ide	One-slit burner 50 mm, coded			
Oxidant	Compressed air and $N_2$ (trous oxide)	D (ni- Inlet pressure: 400 to 600 kPa			
	Nebulizer flow				
	Air	400 to 600 NL/h			
	N <sub>2</sub> O	320 to 480 NL/h			
	Auxiliary oxidant (air or	r N <sub>2</sub> O)			
	Air	3 levels: 75 / 150 / 225 NL/h			
	N2O	3 stages: 60 / 120 / 180 NL/h			
Fuel gas	Acetylene	Inlet pressure: 80 to 160 kPa Consumption: 40 to 315 NL/h			
Nebulizer	Production of the sample aerosol				
	Mode of action	Pneumatic radial clearance nebulizer			
	Material	Platinum-rhodium cannula, PEEK-nozzle			
	Nebulizer	Throughput rate 4 to 6 mL/min			
Siphon	Integrated monitoring o	of the correct filling level (80 mm water column)			
	Mode of action	Float, corrosion proof			
Burner adjustment	Height	4 to 18 mm, automated			
	Depth	Factory preset			
		The novAA 800 D allows a manual adjustment			
	Rotation	0 to 90°, manual			
Safety circuits	Monitoring of	Burner and burner type Fuel gas pressure			
		Inlet pressure oxidant (air and $N_2O$ )			
		Oxidant flow through the nebulizer			
		Siphon filling level			
		Flame			

# 9.1.5 Accessories data

Autosampler AS-GF	Autosampler for adding liquid samples, completely PC-controlled		
	Sample tray	108 positions	
	Sample cups	100 pieces, 1.5 mL	
	Special cups	8 pieces, 5 mL	
	Pipetting volume	1 to 50 μL	
	Rinse volume	0.5 mL, number of wash cycles can be selected	
	Program methods	Standard Modifier Dilution Addition Automatic enrich- ment	
	Mass	7.2 kg	
Autosampler AS-F	Autosampler without dilution function, completely PC-controlled		
	Sample tray 139/15		
	Sample cups	129 pieces, 15 mL	
	Special cups	10 pieces, 50 mL	
	Sample tray 54/ 50		
	Sample cups	54 pieces, 50 mL	
	Power supply	Via AAS basic instrument	
	Wash bottle	2 L	
	Mass	6.5 kg	
Autosampler AS-FD	Autosampler with dilution function, completely PC-controlled		
	Sample tray 139/15		
	Sample cups	129 pieces, 15 mL	
	Special cups	10 pieces, 50 mL	
	Sample tray 54/ 50		
	Sample cups	54 pieces, 50 mL	
	Dosing unit in the Fluidics me	odule 5 mL	
	Power supply	Via AAS basic instrument	
	Wash bottle	2 L	
	Bottle for diluent	2 L	
	Mass (total)	10.0 kg	
	Sampler	6.5 kg	
	Fluidics module	3.5 kg	
Injection module	Model: SFS 6 (Segmented Flow Star), PC-controlled		
	Stable burner conditions ensu	ured by continuous purging and constant temperature conditions	
	Sample volume for individua ysis	anal- 300 μL (minimum volume)	
	Power supply	Via AAS basic instrument	

Mobile cooling unit	Model: KM 5, air cooler with thermostat; CFC-free		
-	Tank capacity	5 L	
	Preset temperature	35 ℃	
-	Capacity	max. 3 L/min	
Piston compressor	Model: PLANET L-S50-15 Standard, Compressed air supply for the flame technique		
	Tank capacity	15 L	
	Dimensions (diameter x heigh	t) 400 mm x 480 mm	
	Power supply	230 V, 50 Hz or	
		230 V, 60 Hz	
	Mass	27 kg	
	Max. operating pressure	800 kPa	
Scraper	Automatic burner head cleaning device for nitrous-oxide flame, PC-controlled		
	Power supply	Via AAS basic instrument	
Air Purge Kit (APK)	Purging of the spectrometer with purified air		
	Dimensions (H x W x D)	245 mm x 265 mm x 260 mm	
	Power supply	100 – 240 V	
		50/60 Hz	
	Power consumption	Max. 15 VA	
	Fuse	2 x T1.6 AH	
	Mass	3.2 kg	
	For further information refer to the operating instructions for the Air Purge Kit (APK).		
Hydride system	Chemical generation of hydride in flow injection and batch mode; devices with modular de- sign for easy adaptation to changing requirements		
	Models	HS 60 modular, HS 55 modular, HS 50	
	Techniques	Hydride technique, mercury-cold-vapor technique and HydrEA	

For further information refer to the operating instructions of the hydride system.

# 9.2 Guidelines and standards

Protection class and protection rating	The novAA 800 has the protection class I. The casing has the protection rating IP 20.
Device safety	The novAA 800 complies with the safety standards
	<ul> <li>DIN EN 61010-1 (VDE 0411T.1; IEC 61010-1)</li> </ul>
	<ul> <li>DIN EN 61010-2-061 (IEC 61010-2-061)</li> </ul>
EMC compatibility	The novAA 800 is tested for suppression of radio interference, noise immunity and interference emission in accordance with class A of the standard DIN EN 55011 and fulfils the respective requirements
	<ul> <li>DIN EN 61326</li> </ul>
Environmental compatibility	The novAA 800 has been tested for environmental compatibility and fulfills the re- quirements stipulated by
	DIN ISO 9022-3:2000
	<ul> <li>DIN ISO 9022-2:2003/01</li> </ul>
Directives for China	The device contains restricted substances (according to directive "Management Meth- ods for the Restriction of the Use of Hazardous Substances in Electrical and Electronic Products"). Analytik Jena guarantees, that those hazardous substances may not leak out during the next 25 years when the device is used in accordance with its intended purpose.
EU directives	The novAA 800 is built and tested according to standards that fulfill the requirements stipulated by the EU directives 2014/35/EU, 2014/30/EU and 2011/65/EU.
	Each device leaves the manufacturer in a pristine and technically safe state. To main- tain this condition and to ensure safe operation, the operator must strictly observe the safety and operating instructions contained in this manual. For accessories which have also been supplied, and system components from other manufacturers, their operating instructions should be referred to.

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