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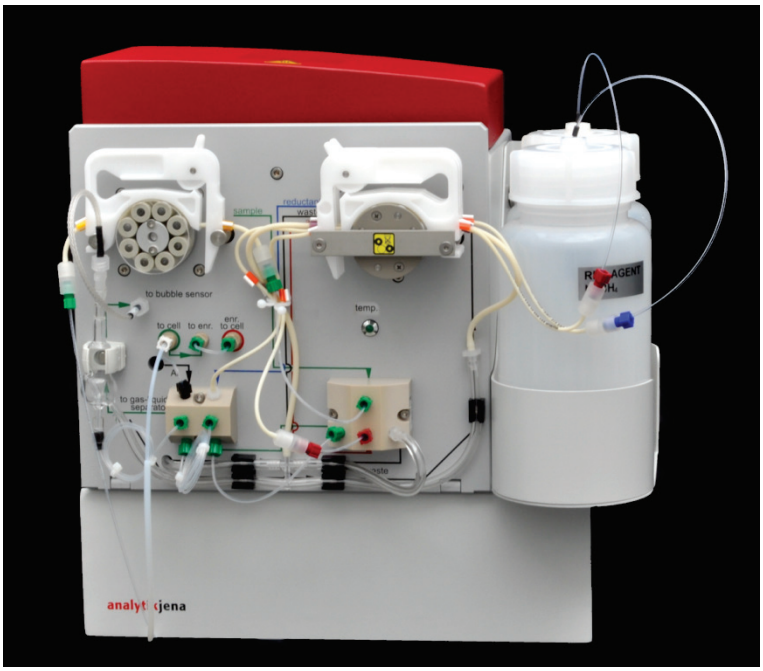
Operating Manual

HS 60 modular
Hg/Hydride System Flow Injection
HydrEA System Flow Injection



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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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Implementation of the Technical Documentation

Analytik Jena GmbH

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1 Basic Information

1.1 User manual notes

The HS 60 modular is intended for operation by qualified specialist personnel observing this user manual.

The user manual informs about the design and operation of the HS 60 modular and provides personnel familiar with analysis the necessary know-how for the safe handling of the equipment and its components. The user manual further includes notes on the maintenance and service of the equipment and potential causes and remedies of any faults.

User manual conventions

Instructions for action which occur in chronological order are numbered and combined into action units and furnished with the corresponding results.

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

Safety notes are indicated by symbols and a signal word. The type and source of the danger are stated together with notes on preventing the danger. The meaning of the symbols and signal words used is explained in the chapter "Safety notes".

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

- ❑ Program terms are identified with SMALL CAPS.
- ❑ Buttons are shown by square brackets (e. g. Button [OK])
- ❑ Menu items are divided by arrows (e. g. FILE ▸ OPEN).

1.2 Intended use

The HS 60 modular must only be used in conjunction with an atomic absorption spectrometer from Analytik Jena. Any departure from the instructions for proper use may lead to warranty restrictions and reduced manufacturer liability in the case of damage.

If the safety instructions are not observed in handling the HS 60 modular, this is taken to be a use which deviates from the intended purpose. Safety instructions are to be found on the equipment itself, in section "Safety instructions” p. 8 and in the description of the relevant work steps.

2 Technical data

Technical data	
Designation/type	HS 60 modular
Methods (dependent on the configuration installed)	Hydride technique Hg cold vapor method without enrichment Hg cold vapor method with enrichment HydrEA method
Operating modes	Flow injection with/without sampler FBR (Fast Baseline Return) method for Hg detection without enrichment (= After reaching the signal maximum the Hg cell is flushed with a greater gas flow.)
detectable elements	As, Bi, Hg, Sb, Se, Sn, Te
Dimensions (W × H × D)	360 × 370 × 240 mm
Mass	approx. 14 kg
Reagents	
Reduction agent (RA)	Sodium borohydride $NaBH_4$ with sodium hydroxide NaOH in a ratio of 3:1 Concentration guide value: 0.3 % $NaBH_4$ + 0.1 % NaOH Tin(II) chloride $SnCl_2$ as alternative for Hg detection
Acid (A)	Hydrochloric acid HCl
Main functional groups	
1-channel hose pump for sample transport	Equipment: Ismaprene hose ID = 1.42 mm; plug: yellow Pumping speed: 4 stages Delivery rate: adjustable from 4–11 mL/min
3-channel hose pump for components	Center: waste exhaust pump hose Ismaprene hose ID = 2.06 mm; plug: purple Rear: Reduction agent Ismaprene hose ID = 0.89 mm; plug: orange Front: Acid Ismaprene hose ID = 0.89 mm; plug: orange Pumping speed: adjusted to sample pump Delivery rate: adjustable from 1-7 mL/min
Reaction unit	PEEK reactor with 120° angle of incidence between sample/acid and reduction agent and reaction products and argon flow and 0.75 m hose loop
"Hg Plus" module	Gold collector: 0.5 g gold/platinum alloy AuPt 10 as fine mesh Bakeout temperature: 630 °C controlled Cooling: axial fan
Technical data	
Cell unit	Heating: electric, temperature for hydride generating elements: 600 °C to 950 °C Temperature for Hg: room temperature or 150 °C Temperature consistency: ±10 °C of target temperature

Absorption cells	Quartz cells with removable quartz windows: Length 140 mm, ID 15 mm Hg cell: length 200 mm
Inert gas Argon	Purity: min. 99.999 Vol.% Inlet pressure: 600 kPa Operating pressure: 150 kPa Gas flow: FBR gas flow F2: 20 L/h, transport and flushing gas: F3: 6 L/h; F4: 25 L/h; F3+F4: 31 L/h
Operating times F.I. operation	Sample load time: Time during which the sample pump loads the intake hose up to the two-valve assembly with sample. Reaction time: Time during which the sample pump pumps sample into the reactor. AZ wait time: Waiting time immediately prior to zero balance Wash time 1, 2, 3: Times for the transport of the reaction gas with the Argon flow on various gas paths Heat. time collector: Time during which the heating of the gold collector is activated. Cool. time collector: Time during which the fan of the gold collector is activated.
Electrical variables	
Power supply	dependent on base module: 220-230 V or 100-110 V
Frequency	50/60 Hz
Protection	G fuse sets (5 × 20 mm) according to EC 60127/250V Fuse F1/F2: T3,15 A/H for 230 V, T6,3 A/H for 110 V
Power consumption during heating	650 VA
Power consumption during continuous operation	400 VA
Overvoltage category	II according to DIN EN 61010-1
Contamination category	2 according to DIN EN 61010-1
Safety class	I
Protection type	IP 20
Environmental conditions	
Temperature during storage and transport	-40 °C to +50 °C according to DIN 58390-2
Temperature during operation	+10 °C to +35 °C
Humidity	max. 90 % at +30 °C
Corrosion protection	corrosion-resistant towards the analysis samples
Recommended max. operating altitude	2000 m

3 Safety instructions

3.1 General notes

For your own safety and to ensure error-free and safe operation of the HS 60 modular, please read this chapter carefully before commissioning.

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

IMPORTANT

The separate user manual points out specific hazards that may arise when working with the AAS device.

3.2 Standards and directives

The HS 60 modular was manufactured according to the currently valid technology regulations and the approved safety related regulations.

When constructing the device the relevant safety and health requirements of the applicable laws, standards and regulations were applied. The safety of the device is confirmed by the CE mark and the declaration of conformity.

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

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

Warnings regarding potential danger do not replace work protection regulations!

3.3 Symbols and signal words used

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

WARNING

Indicates a potentially hazardous situation.

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

CAUTION

Indicates a potentially hazardous situation.

If it is not prevented light or minor injuries and material damage can result.

ATTENTION

Indicates a potentially hazardous situation which, unless avoided, may cause damage to the device or items in its vicinity.

IMPORTANT

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

3.4 Safety labeling at the HS 60 modular

Safety symbols have been attached to the HS 60 modular and accessories whose content must always be observed.

Damaged or missing safety symbols can cause incorrect actions leading to personal injury or material damage! The safety symbols must not be removed! Damaged safety symbols must be replaced without delay!

The following safety symbols have been attached to the HS 60 modular and accessories:

General warning

Warning against hot surface

Caution! Prior to assembly or removal and opening the device disconnect the mains plug.

Warning against crushing

3.5 Technical condition

The HS 60 modular corresponds in its design and construction to the current state of the art technology. Unauthorized modifications or changes, especially such that affect the safety of the staff and the environment, are generally not allowed.

Observe the following notes:

- ❑ Any manipulation of the safety equipment is prohibited!
- ❑ In case of an accident manipulations of the safety equipment will be interpreted as deliberate!
- ❑ The operator must only operate the device in a sound and operationally safe condition. The technical condition must always comply with the legal requirements and regulations.
- ❑ Prior to every use the device must be checked for damage and sound condition.
- ❑ Any changes in the device affecting its safety must be reported by the operating personnel to the operator without delay.

- ❑ The equipment components must only be connected to supply cables intended and designed for this purpose.
- ❑ All safety equipment and interlocks must be well accessible and regularly checked for proper operation.

3.6 Requirements for the operating personnel

The HS 60 modular 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 add-on devices.

The HS 60 modular may pose dangers if it is not used by trained personnel, improperly or other than intended.

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

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

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

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

Observe the following notes:

- ❑ The device must only be commissioned, operated and serviced by trained personnel instructed in technical safety.
- ❑ The operation or servicing of the device by minors or individuals under the influence of alcohol, drugs or medication is not permitted.
- ❑ It must be ensured that only authorized personnel works at the device.
- ❑ The operating personnel must be familiar with the dangers arising from samples to be analyzed and the auxiliary and operating materials used. The appropriate protective equipment must be used.
- ❑ Prior to pauses or at the end of the work appropriate skin cleaning and protection measures must be carried out.
- ❑ Eating, drinking, smoking or handling open flames in the operating room of the Hg hydride system is prohibited!

3.7 Safety instructions, transport and installation

The HS 60 modular is always installed by the customer service department of Analytik Jena or its authorized and trained specialist personnel.

Independent assembly and installation are not permitted. Incorrect installation can create serious hazards.

Observe the following notes:

- ❑ Insufficiently secured components pose a risk of injury! During transport the components of the equipment must be secured in accordance with the instructions in the user manual.
- ❑ Only transport the device in its original packaging! Ensure that all modules are securely connected to each other and the device is completely empty. Flush the pump and metering hoses thoroughly to prevent reduction agent solution or acid from dripping out. The solutions are aggressive and attack clothing.
- ❑ To prevent health damage the following must be observed when moving the device in the laboratory (lifting and carrying):
  - The Hg/Hydride system has a mass of approx. 14 kg. Since the device does not have handles, grip the device firmly with both hands at the continuous board of the basic module.
  - The guide values and statutory limits for lifting and carrying loads without auxiliary equipment must be observed and adhered to.

3.8 Safety instructions - operation

3.8.1 General

The operator of the HS 60 modular must make sure before each commissioning that the condition of the device including the safety equipment is sound. This applies in particular after each modification or extension of the device or its repair.

Observe the following notes:

- ❑ The device must only be operated if all module and protective devices (e. g. covers) are present, properly installed and fully operational.
- ❑ The sound condition of the protection and safety equipment must be checked regularly. Any defects must be corrected as soon as they occur.
- ❑ Protective and safety equipment must never be removed, modified or decommissioned during operation.
- ❑ Free access to the mains switch on the ride side panel must be ensured 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.

- ❑ The ventilation equipment on the device must be in good working condition. Covered vents or ventilation slits etc. may cause the device to break down or may cause damage to it.
- ❑ During the commissioning of the device there is a risk of the cell unit corroding in the acid residue remaining in the siphon. The siphon in the AAS device should therefore be flushed with 0.5 L water via the mixing chamber connection before placing the cell unit onto the mixing chamber connection.
- ❑ High temperatures arise with a heated cell unit. Hot components must not be touched during or directly after the operation of the device. The cooling-down times to room temperature (1 h) must be observed.
- ❑ Caution when handling glass components. Risk of broken glass and therefore risk of injury!
- ❑ Flammable materials must be kept away from the cell unit.

3.8.2 Safety instructions - Protection against explosion and fire

The HS 60 modular may not be operated in an explosive environment. Smoking or handling open flames in the operating room of the Hg/Hydride system is prohibited!

The operating personnel must be familiar with the location of the fire-fighting equipment in the operating room of the device.

3.8.3 Safety instructions - electrical equipment

Work on electrical components of the HS 60 modular may only be carried out by a qualified electrician in accordance with the applicable electrical engineering rules. The device is supplied with mains voltage. Lethal voltages may occur in the process!

Observe the following notes:

- ❑ The HS 60 modular must always be connected to and disconnected from the mains whilst switched off. For the connection the multiple socket supplied with the AAS device must be used.
- ❑ Removal of the device cover of the basic module may only be carried out by the customer service of Analytik Jena and specially authorized technicians.
- ❑ For electrical work the mains plug must be disconnected from the mains outlet.
- ❑ The electrical components must be checked regularly by a qualified electrician. Any defects, such as loose connections, faulty or damaged cables, must be repaired without delay.
- ❑ The device must be switched off immediately at the device switch (right side panel) and the mains plug disconnected if there is any interference with the electric components.

3.8.4 Safety instructions for compressed gas containers and systems

The inert gas (Argon) is taken from compressed gas containers or local compressed gas systems. The required purity of the carrier gas must be ensured (see chapter 2" Technical data" p.6)!

Work on compressed gas containers and systems must only be carried out by individuals with specialist knowledge and experience in compressed gas systems.

Observe the following notes:

- ❑ For gas cylinder or gas plant operation, the safety instructions and guidelines which are valid at the operating location must be strictly complied with.
- ❑ High pressure hoses and pressure reducers may only be used for the assigned gases.
- ❑ All pipes, hoses and screw connections must be checked regularly for leaks and externally visible damage. Leaks and damaged must be repaired without delay.
- ❑ Prior to inspection, maintenance and repair the gas supply must be closed.
- ❑ After successful repair and service of the components of the compressed air containers or system the HS 60 modular must be checked for sound operation prior to recommissioning!
- ❑ Independent assembly and installation are not permitted.

3.8.5 Handling of auxiliary and operating materials

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

When handling dangerous substances local safety codes and guidelines must be observed.

The following general notes do not replace the specific local regulations or the regulations in the EC safety data sheets of the manufacturers for the auxiliary and operating materials.

Observe the following notes:

- ❑ The HS 60 modular must only be used in conjunction with the AAS device under an active gas extractor.
- ❑ The relevant regulations and the notes in the EC safety data sheets of the manufacturers have to be observed and complied with regards to storage, handing, use and disposal for all auxiliary and operation materials used during operation or maintenance of the HS 60 modular.
- ❑ Auxiliary and operation materials may never be placed in containers or vessels for food. The approved containers for the relevant material are to be used and these have to be labeled accordingly. The notes on the labels have to be observed!

- ❑ Protective goggles and rubber gloves have to be worn when handing reagents.
  - – Sodium borohydride ($NaBH_4$) and sodium hydroxide are strongly corrosive, hygroscopic and, in solution, extremely aggressive. Dripping off and splashing of the reduction solution must be avoided.
  - – Cleaning with hydrofluoric acid and concentrated hydrochloric acid must take place under an extractor. Personal protective equipment (rubber apron, gloves and face mask) must be worn.
- ❑ Biological samples have to be handled according to local guidelines regarding the handling of infectious material.
- ❑ Auxiliary and operating materials as well as their containers may not be disposed in domestic waste or enter the sewage system or the soil. The residual fluid from the Hg/Hydride system and the sampler must be collected in the resistant 10 L bottle included in the scope of delivery of the AAS device. The applicable regulations for the disposal of the residual substances must be meticulously observed.
- ❑ When measuring cyanide-containing material, ensure that prussic acid cannot be generated in the waste bottle.
- ❑ Ensure good room ventilation in working rooms.

WARNING

Hydrogen is released by the reaction of sodium borohydride with the acidic sample solution. The formation of a hot, explosive hydrogen-air mixture in the cells must be excluded. The gas lines from the reaction cup to the cell outlet must be kept oxygen-free. To this end take the following measures:

- ❑ Always ensure that the cells with the windows are closed and gas-tight. Even with a small leak at the end faces, the cell must be replaced.
- ❑ Direct the gas from the cell outlet to the exhaust unit.

3.8.6 Safety instructions - service and repair

The Hg/Hydride system is usually serviced by the customer service department of Analytik Jena or its authorized and trained specialist personnel.

Independent servicing can maladjust or damage the device. Therefore, the operator may generally only carry out the tasks listed in chapter "Maintenance and care".

Observe the following notes:

- ❑ The exterior of the Hg/Hydride system may only be cleaned with a damp, not dripping, cloth after the device has been switched off.
- ❑ Any service and repair work at the device may usually only be carried out in the switched-off condition (unless stated otherwise).
- ❑ Service tasks and the replacement of system components (e. g. removal of the cell) must only be performed after an adequate cooling down phase.

- ❑ Prior to servicing or repair the energy and gas supplies must be disconnected and the device must be vented!

Only use original accessories and spare parts from Analytik Jena. The notes in chapter "Maintenance and care" p. 45 must be observed.

- ❑ All protective equipment must be reinstalled correctly immediately after completion of the service and repair work and be checked for operation!

- ❑ After replacing the functional module (batch/flow injection) the Hg/Hydride system may only be put back into operation if the new functional module has been properly screwed to the basic module.

3.9 Behavior during emergencies

- ❑ If there is no immediate danger of injury, immediately switch of the mains switch at the right-hand side panel and/or disconnect the mains plug from the mains outlet during dangerous situations or accidents! Close the gas supply as soon as possible after switching off the device.

Because a rapid response can save lives during an emergency, the following has to be ensured:

- ❑ The operating staff must be familiar with the location of safety equipment, accident and danger alarms as well as first aid and rescue equipment as well as their handling.

- ❑ The operator is responsible for the respective training of the operating staff.

- ❑ All equipment for first aid (first-aid kit, eyewash bottles, stretcher, etc.) as well as equipment for firefighting (fire extinguishers) must be within reach and easy to access. All equipment has to be in a sound condition and should be checked at regular intervals.

4 Technical description

4.1 Methods and overview of the Hg/Hydride systems

The hydride method

The hydride method enables the matrix-free detection of the elements As, Bi, Sb, Se, Sn and Te. It is based on the generation of gaseous metal hydrides through the reduction of acidic samples with sodium borohydride $NaBH_4$. The metal hydrides are transported by the carrier gas and the released hydrogen to the quartz cell. There they gradually decompose through impact processes with gas particles and the glass wall at temperatures from 850 °C to 950 °C. The free metal atoms absorb the primary radiation on the resonance line.

With the hydride method spectral interference is practically eliminated, because only the element to be detected reaches the atomizer as gaseous metal hydride.

The cold vapor method

The cold vapor method is used to detect mercury. In addition to sodium borohydride $NaBH_4$ tin(II) chloride $SnCl_2$ is also used as reduction agent. During the reaction with the acidic sample solution atomic Hg vapor is generated which is transported by the carrier gas Argon to the Hg cell. The free Hg atoms absorb the primary radiation on the resonance line. The heating of the cell from room temperature to 150 °C reduces background interference due to moisture.

The HydrEA method

The HydrEA method combines the hydride or Hg cold vapor method with the graphite tube method. It assists the highly sensitive selective detection of the hydride-generating elements As, Bi, Sb, Se, Sn and Te and of Hg with the electrothermal atomizer.

The Hg/Hydride system generates the gaseous metal hydrides or the atomic Hg vapor. The sampler graphite (AS-GF) transfers these with the carrier gas Argon to the graphite tube furnace. There they enrich at a preheating temperature of 300 °C on the iridium or of 65 °C on the gold-coated standard tube for wall atomization. At temperatures of 2100 °C or 950 °C the deposited metal hydrides or Hg atoms atomize. The generated atom vapor cloud absorbs the primary radiation on the resonance line.

Overview of the Hg/Hydride systems

The Hg/Hydride systems available range from the simple batch systems for users with small samples through to fully automated flow injection devices.

HS 50:	basic batch system with pneumatic principle of operation. 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 Hg detection. The reduction agent solution is metered by a 1-channel hose pump.
HS 60 modular:	Hg/Hydride system for flow injection operation with electrically heated cell unit with or without "Hg Plus" module for Hg detection.

The Hg/Hydride systems can be used independent of the equipment level for the methods described above.

The Hg/Hydride system HS 60 modular

The HS 60 modular consists of the basic module, the flow injection functional module and the "Hg Plus" module as optional accessory. The three modules are plugged on top of each other and electrically connected through mixed plug connectors. They can be swapped or retrofitted unassisted by the user.

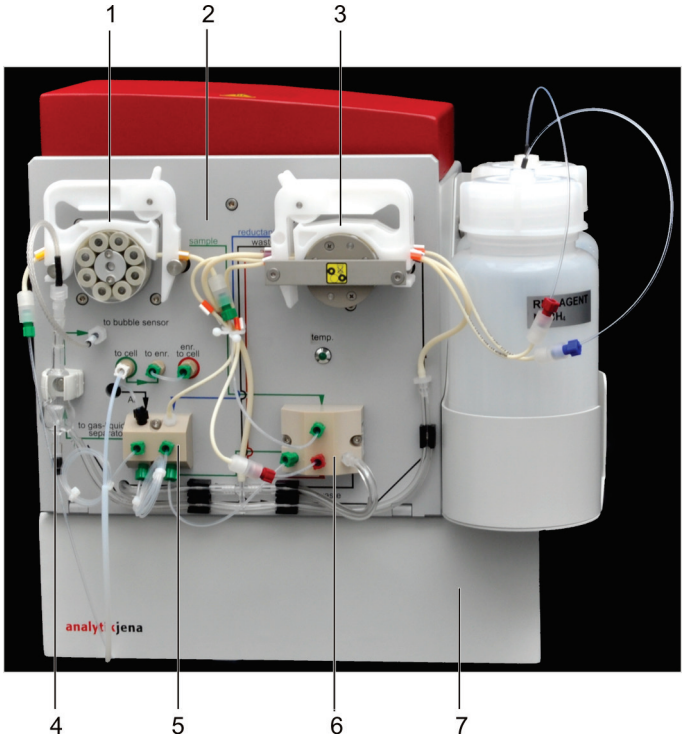
The HS 60 modular can be used with the following AAs devices:

- ZEEnit 700 P / ZEEnit 650 P
- novAA 400 P / novAA 350
- contrAA 700 / contrAA 600 / contrAA 300
- Earlier devices with RS 232 interface

Figure 1 HS 60 modular with AAS novAA 400 P

At the front plate of the flow injection functional module the following assemblies are located:

- 1-channel hose pump for sample transport
- 3-channel hose pump for the transport of waste, reduction agent and acid
- 2-valve group to switch from acid to sample
- Reactor with reaction loop
- Gas/fluid separator to separate the gaseous reaction products from the remaining fluid



1 sample pump (1-channel hose pump)
2 flow injection functional module
3 component pump (3-channel hose pump)
4 gas/fluid separator
5 reactor
6 2-valve group
7 basic module

Figure 2 HS 60 modular (front view)

All important assemblies and the pump and connection hoses are located at the front plate of the functional module. The hoses are easily accessible and can be replaced by the user. The colored line on the front plate identifies the hose routing and thereby facilitates maintenance.

On the right-hand side of the device are the storage bottles for the reduction agent and the acid in a holder. Here the electrical connections are located.

The following are located in the interior of the functional module:

- ❑ Bubble sensor with change-over valve to monitor the reaction gas for moisture
- ❑ 4-valve group for the gas supply

The "Hg Plus" module is inserted from the top into the flow injection functional module and connected to it electrically. The hoses are routed to the frame of the functional module and from there to the front plate. When changing between the operating modes hydride/Hg without enrichment and Hg with enrichment the user only changes the hose routing at the front plate.

4.2 Basic system design

The HS 60 modular generally works with sodium borohydride $NaBH_4$ as reduction agent; for Hg detection tin(II) chloride $SnCl_2$ can also be used. Argon is used as carrier and flushing gas.

ATTENTION

Changing the reduction agent requires major maintenance work. All hoses having come into contact with the reduction agent must be replaced and the system flushed thoroughly.

The sample solution is aspired by a 1-channel hose pump; the 3-channel hose pump transports the acid and reduction agent. A 2-valve group optionally switches sample or acid onto the reactor and the respective other component onto waste. In the reactor the sample and reduction agent meet, the sample is reduced and gaseous metal hydride or atomic Hg vapor is released. In addition, hydrogen is released. The gaseous reaction products are captured by the Argon flow and transported to the gas/fluid separator. Here the gas phase (metal hydride or Hg vapor, Argon and hydrogen) and the fluid phase are separated. The residual fluid is pumped away by the 3-channel hose pump.

The separated gas is either passed directly into the quartz cell and measured in absorption or directed for Hg enrichment via a gold collector. The enriched mercury is released during the bakeout of the gold collector and transported to the cell by the Argon flow directly connected to one of the 4-valve groups.

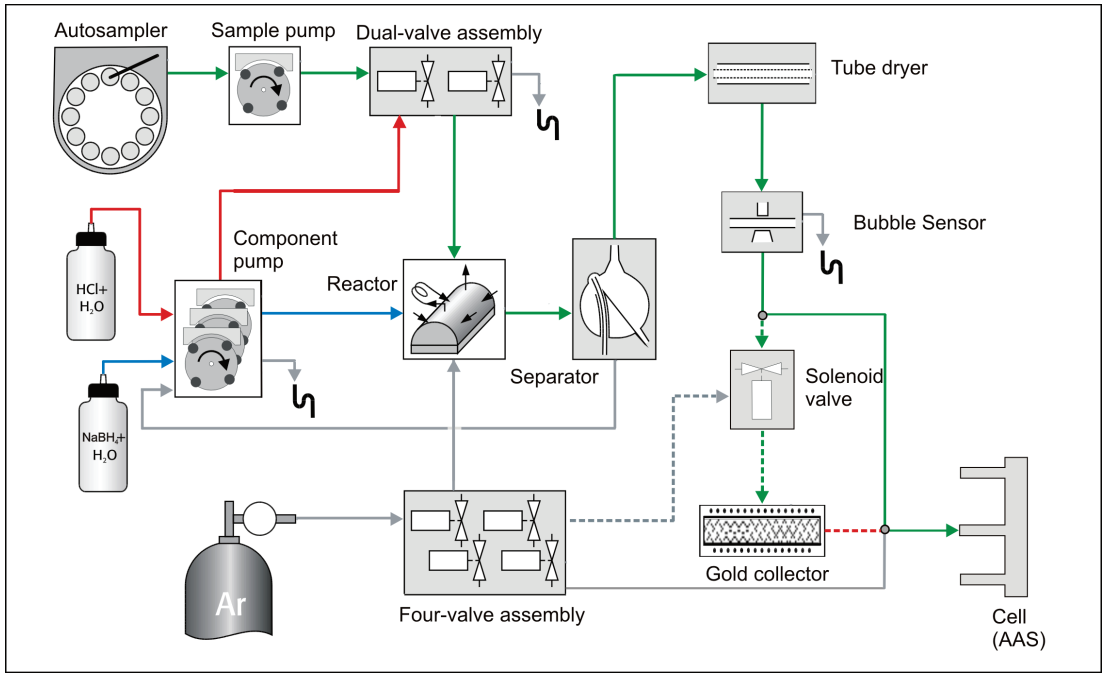


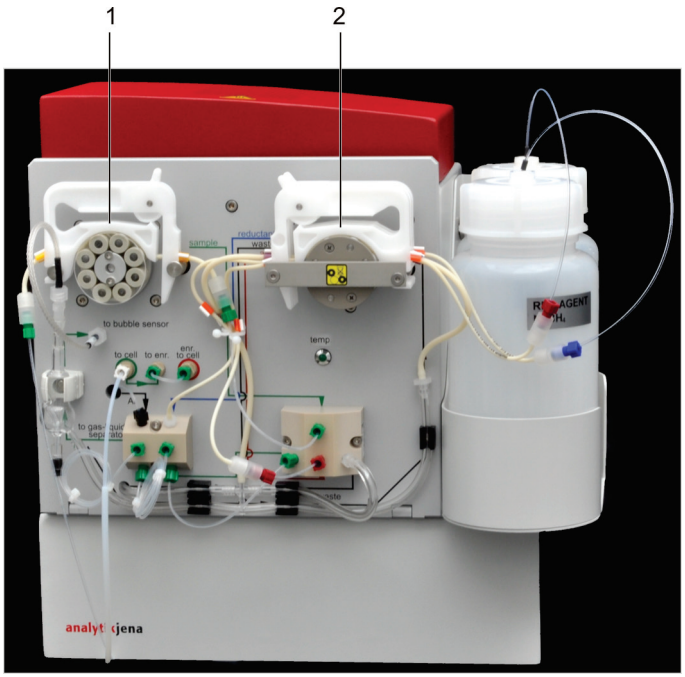
Figure 3 Functional diagram of the HS 60 modular

IMPORTANT

Information on the technical design of the AAS device can be found in the separate user manual.

4.3 Design of the Hg/Hydride system HS 60 modular

4.3.1 Hose pumps



1 sample pump (1-channel hose pump)

2 component pump (3-channel hose pump)

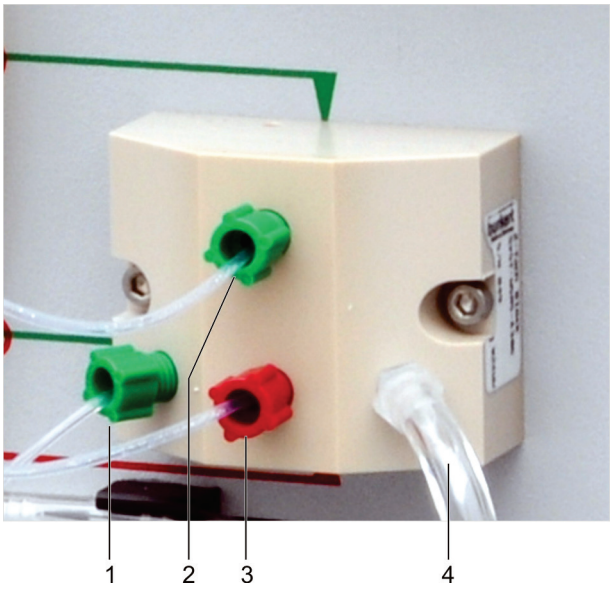
Figure 4 Hose pumps

The hose pumps are equipped with adjustable snap-in cartridges. The 1-channel pump (1), equipped with an Ismaprene hose of 1.42 mm inside diameter, conveys the sample in 4 selectable velocity stages with pump rates of 5 to 11 mL/min. The sample pump is only running during the loading and reaction time.

The 3-channel hose pump (2) conveys acid and reduction agent with the front and rear channel and pumps off the fluid phase from the gas/fluid separator with the center channel. During measurements of metal hydrides and mercury without enrichment the 3-channel pump is running throughout the entire measuring cycle, during measurements of mercury with enrichment it is running from the start of the measuring cycle up to the end of the flushing time 1 with a fixed pump velocity. During the reaction phase the pump velocity is adapted internally to the velocity of the sample pump. The delivery rate for reduction agent and acid of 1–7 mL/min. is sufficient.

Pump hose overview		
Function	Stopper	ID [mm]
3-channel pump		
reduction agent hose	orange/orange	0.89
acid hose	orange/orange	0.89
exhaust pump hose	purple/purple	2.06
1-channel pump		
sample hose	yellow/yellow	1.42

4.3.2 The 2-valve group

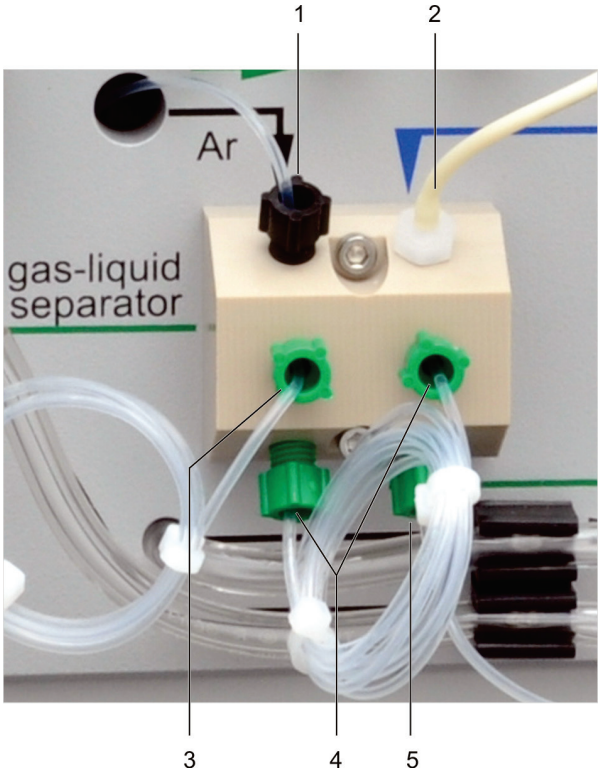


1 sample to the reactor
2 sample from the sample pump
3 acid from the component pump
4 to waste

Figure 5 2-valve group

The 2-valve group consists of 2 inert solenoid valves on a PEEK body. During the reaction phase it switches the sample flow to the reactor and the acid to waste. In the basic state the acid flow is switched to the reactor and the sample flows to waste with an active sample pump.

4.3.3 Reactor

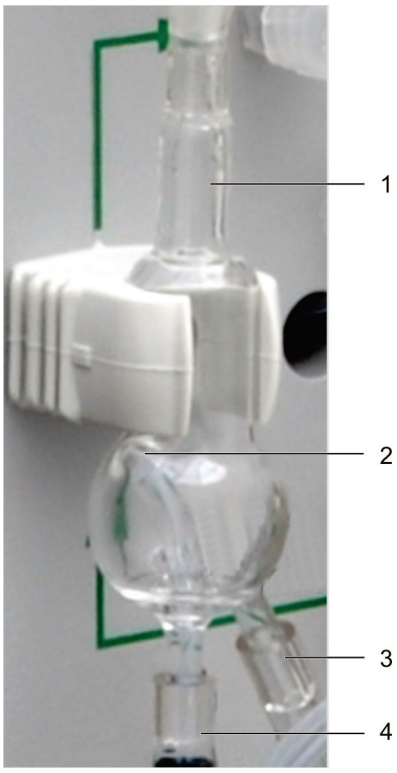


1 Argon inert gas inlet
2 reduction agent inlet
3 outlet of reaction products to the gas/fluid separator
4 hose bridge connections, reactor hose
5 sample inlet (or acid)

Figure 6 Reactor

In the PEEK reactor the sample or acid and the reduction agent meet at an angle of 120° and react to each other. The reacting components are picked up at an angle of 60° to both inlets. The subsequent reaction takes place In the 75 cm long wound MFA hose with 1 mm inside diameter. At a second impact point the Argon flow and reaction products meet at 120° and are discharged at 60°. The reaction, i.e. the release of the gaseous metal hydrides or the atomic mercury is completed in the reactor.

4.3.4 Gas/fluid separator



1 reaction gas outlet
2 bulge
3 fluid waste outlet
4 reaction gas inlet

Figure 7 Gas/fluid separator

The gas/fluid separator made from Duran glass is characterized by a low dead volume. The reaction products are introduced from below (4), the hose terminates in a semi-spherical bulge (2) which reduces the formation of bubbles even with foaming samples significantly. The gaseous reaction products exit towards the top with the Argon carrier gas (1). This reduces the danger of drops being carried along to a minimum. The residual fluid is pumped off from the floor of the gas/fluid separator (3).

4.3.5 Hose membrane dryer

In the hose membrane dryer the reaction gas is dried through moisture exchange with the surrounding air. This removes the residual moisture from the measuring gas. The hose dryer connects the gas/fluid separator to the bubble sensor.

4.3.6 Bubble sensor with change-over valve

The bubble sensor responds to the minutest bubbles and drops in the MFA hose. The fluid causes a change of the refraction index in the MFA hose which is detected by a light barrier. If the bubble sensor is triggered the downstream solenoid valve switches from the direct pass-through to waste and thus prevents the entry of of moisture into the hoses leading to the cell or the gold collector.

4.3.7 4-valve group for gas control

The 4-valve group provides fixed gas flows controlled by the software:

Valve MV2: F2 with 20 L/h as direct gas flow to the cell for the signal cancellation during the FBR method for Hg detection without enrichment

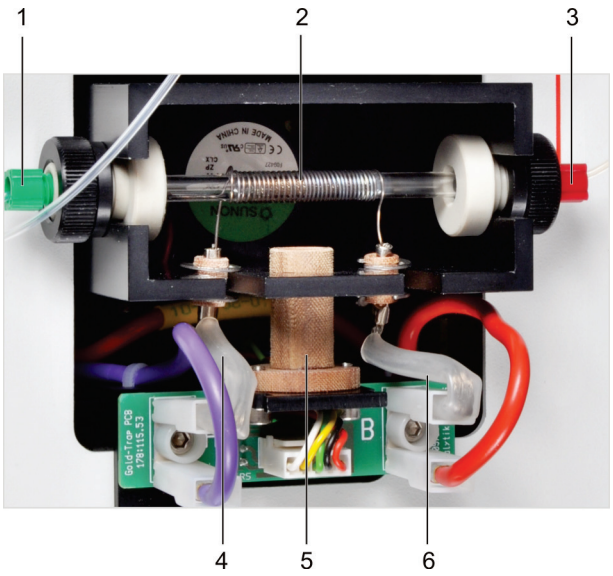
Valve MV3/MV4: F3 with 6 L/h and F4 with 25 L/h combined as transport gas flow.

Valve MV5: switches F3, F4 optionally to the reactor or the gold collector (to expel the released mercury). The gas pressure present is continually monitored by a pressure monitor.

4.3.8 "Hg Plus" module

The "Hg Plus" module is an optional accessory and can be retrofitted by the user. It is located at the top of the functional module. The module comprises in addition to the compartment with the gold collector, sensor and fan a 3/2-way solenoid valve on the inlet side. This solenoid valve switches optionally the reaction gas for loading and the direct gas flow for heating to the gold collector.

The "Hg Plus" module includes as core a loosely rolled up gold/platinum mesh of approx. 20 mm width located and secured in a quartz tube. The gold collector withdraws the free Hg atoms from the passing reaction gas, enriches them on the gold surface and emits them again only after bakeout to approx. 630 °C. The heat is introduced from the outside by a surrounding heating coil. An infrared sensor monitors the bakeout temperature. The gold collector is cooled down by the air flow from an axial fan after bakeout.



1 supply hose (gas inlet)
2 heating wire
3 discharge hose (gas outlet)
4 heating connection
5 infrared sensor
6 heating connection

Figure 8 gold collector

4.4 Measuring processes

IMPORTANT

The measurements are controlled via the software of the AAS device. Information about performing the measurements can be found in the separate user manual of the AAS device. The measuring parameters for the individual elements (hydride formers and Hg) have been shown in the recipes.

4.4.1 Flow injection operation without enrichment and FBR

The first measurement of a calibration or each individual sample measurement starts with the loading phase where the sample path up to the 2-valve group is loaded with a sample. For subsequent measurements of the same sample this phase is omitted.

During the entire measuring process reagents are conveyed by the 3-channel pump. During the auto zero wait time (AZ wait time) constant conditions arise in the cell with constant gas flow to allow the zero value to be determined (AZ).

During the reaction time the 2-valve group switches the sample to the reactor, the sample pump is running. With the start of the reaction the measurement is also started. Reaction time and pump velocity determine the sample volume being processed. During the subsequent flushing time (wash time 1) the system is flushed clear of reaction gas (metal hydride, Hg vapor) by the constant gas flow.

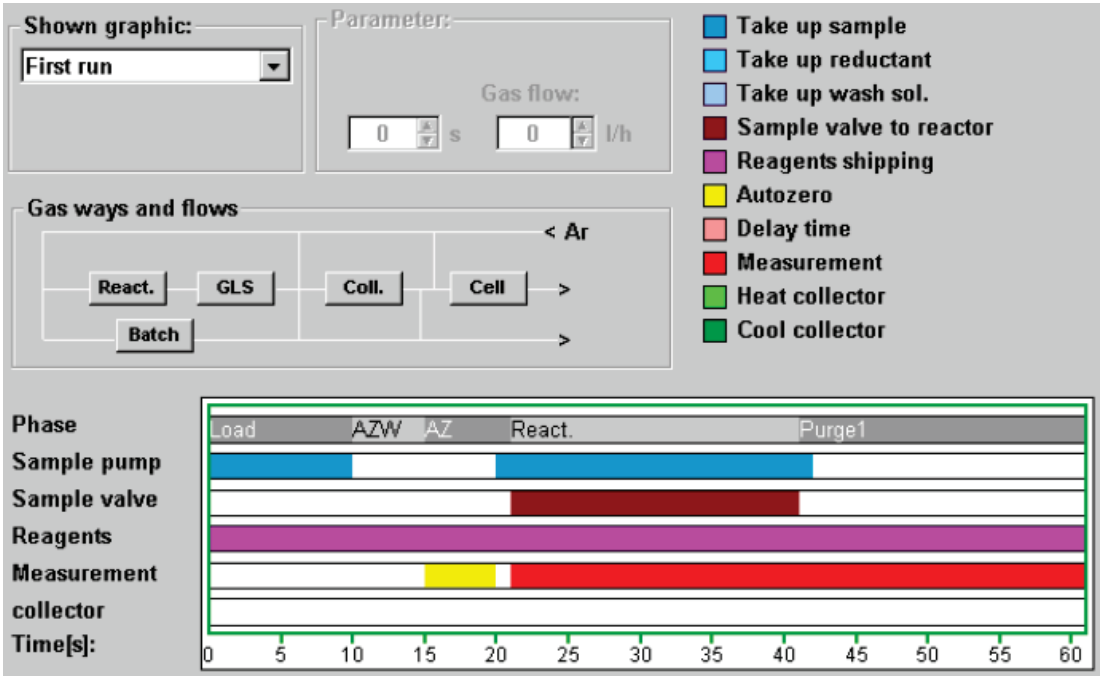


Figure 9 Operation without enrichment and without FBR

4.4.2 Flow injection operation without enrichment, with FBR (fast baseline return)

At the start of the first measurement of a calibration or each individual sample measurement the sample path is loaded with sample up to the 2-valve group (load time). For subsequent measurements of the same sample this phase is omitted. During the auto zero wait time (AZ wait time) a gas flow of 20 L/h flows from the valve MV2 directly to the cell and creates the same conditions for the zero value measurement (AZ) as for the signal cancellation. The freely selectable gas flow through the reaction path simultaneously flows to the waste/exhaust gas.

The 3-channel pump conveys reagents throughout the entire measuring process. During the reaction time the 2-valve group releases the sample to the reactor. Reaction time and sample pump velocity determine the sample volume being processed. The gas flow of 20 L/h is interrupted. Instead the freely selectable gas flow transports the reaction products to the cell.

During the flushing time 1 (wash time 1) the gas flow through the reaction path is maintained.

During the flushing time 2 (wash time 2) the direct gas flow of 20 L/h flushes the cell clear and allows the signal to return quickly to the baseline (Fast Baseline Return, FBR). The freely selectable gas flow through the reaction path flows to the waste/exhaust gas during the flushing time 2 and flushes the system clear.

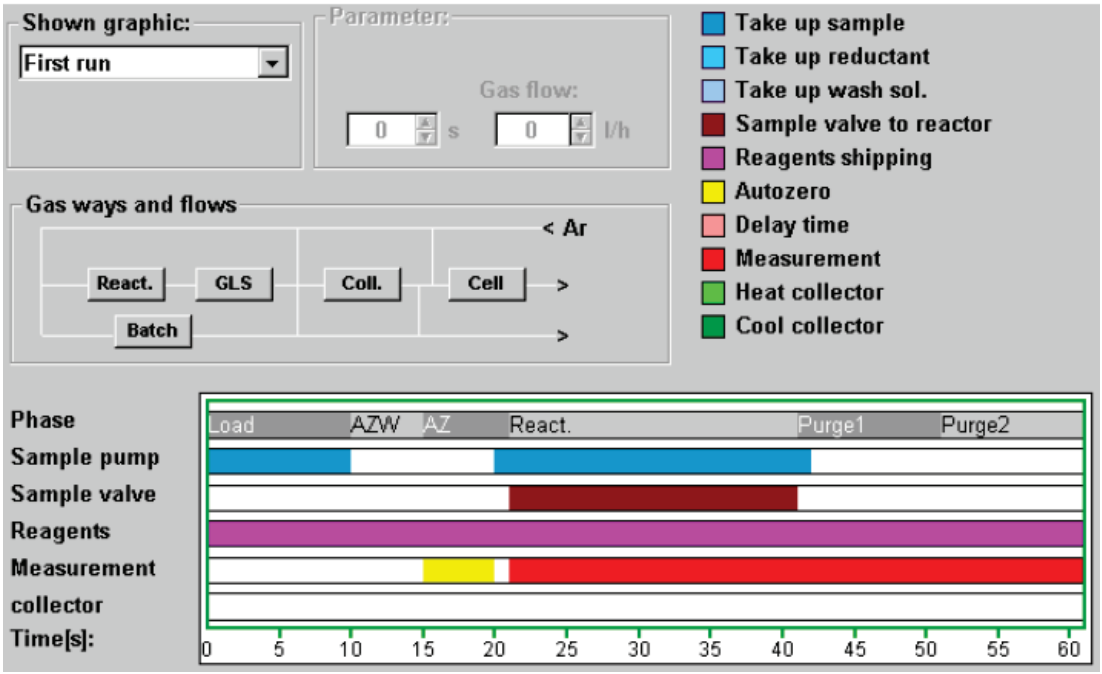


Figure 10 Operation without enrichment with FBR

4.4.3 Flow injection operation with enrichment

At the start of the first measurement of a calibration or each individual sample measurement the sample path is loaded with sample up to the 2-valve group (load time). For subsequent measurements of the same sample this phase is omitted. During the reaction time the sample pump continues running, the 2-valve group switches the sample to the reactor. At the same time the 3-channel pump conveys the reagents. Reaction time and sample pump velocity determine the sample volume being processed. During the reaction time and subsequent flushing time 1 (wash time 1) the gas flow through the reactor transports the released mercury to the gold collector where it is enriched. The 3-channel pump runs up to the end of the flushing time 1.

During the AZ wait time the freely selectable gas flow is directed by the change-over valve MV5 of the 4-valve group directly via the gold collector to the cell. It creates constant conditions for the zero value measurement (AZ).

The bakeout of the gold collector, the flushing time 2 and the measurement are started at the same time. The transport gas flow continues flowing with a freely selectable flow rate directly to the gold collector and transports the released mercury to the cell.

The bakeout of the gold collector is followed by cooling to room temperature.

Flushing time 2 is followed by flushing time 3. A constant gas flow of 31 L/h is flowing. Cell and gold collector are flushed clear; the measuring signal fades out on the baseline.

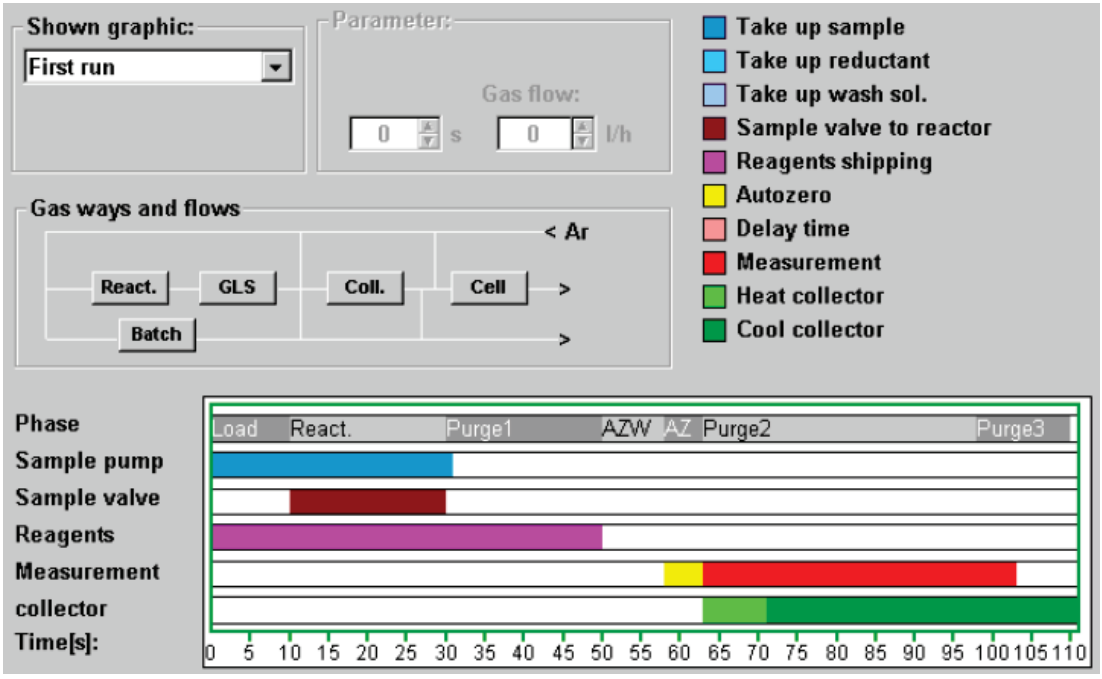


Figure 11 Operation with enrichment

4.4.4 System flushing

System flushing can take place, dependent on the measuring task, at different times:

- ❑ after each sample measurement
- ❑ as action in the sample table
- ❑ only if concentrations have been exceeded

System flushing takes place as selected with diluted acid only or with reduction agent and diluted acid.

When flushing with diluted acid the sample aspiration hose is immersed by the sampler in the flushing container, the flushing pump conveys the acid from the storage bottle into the flushing container. When working without sampler, the sample aspiration hose must be immersed manually in a storage bottle with acid (in line with instructions). During the first half of the flushing time the 2-valve group switches the sample path with acid flow to waste, during the second half to the reactor.

Flushing with reduction agent and diluted acid always starts with reduction agent.

The sample aspiration hose is immersed in a sample container with reduction agent, either automatically by the sampler or, when working without sampler, manually (in line with instructions).

During the first half reduction agent flushing time the 2-valve group switches the sample path with reduction agent flow to waste, during the second half to the reactor.

After completion of the reduction agent flushing time a dwell time of 10 seconds to up to a few minutes may occur. During the dwell time the sample hose, 2-valve group, reactor and gas/fluid separator are exposed to the effects of the reduction agent solution.

After the dwell time the sample aspiration hose is immersed in acid. During the first half of the flushing time the 2-valve group again switches the acid conveyed by the sample pump to waste, during the second half to the reactor.

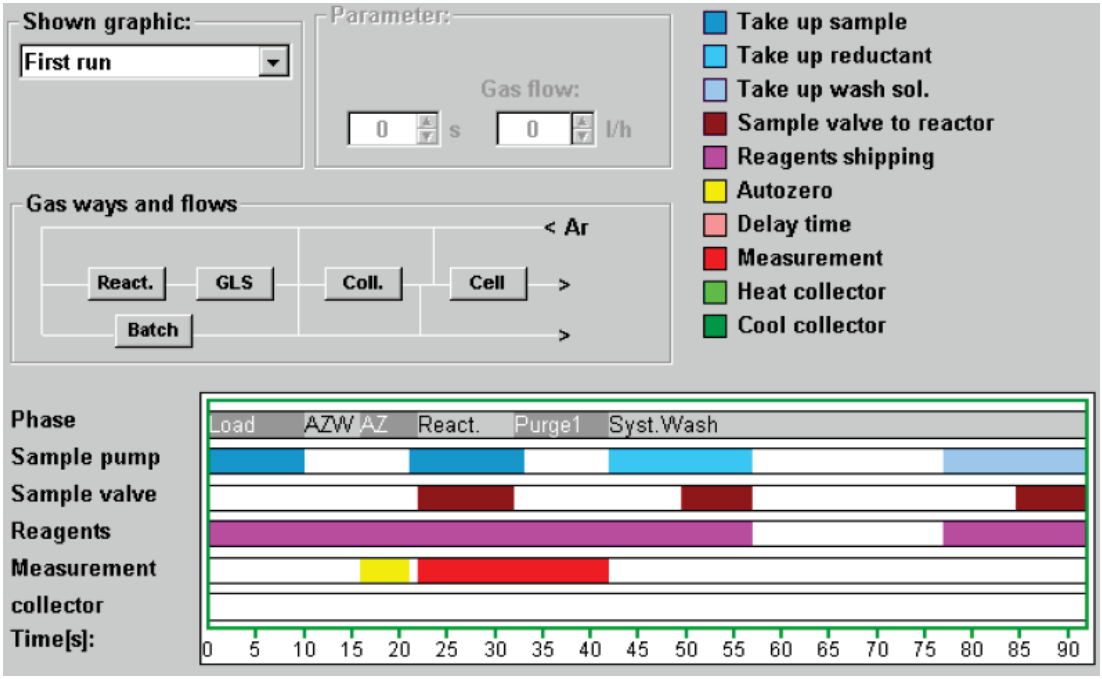


Figure 12 System flushing

5 Commissioning

5.1 Installation and transport conditions

The Hg/Hydride system is normally installed together with the AAS device by the customer service of Analytik Jena or by persons authorized by Analytik Jena. As a subsequent delivery it can also be installed by the operator personnel.

The operator is responsible for everything (e.g., parts, guidelines) which are not included in the original delivery, but which are necessary for operation of the Hg/Hydride system. Operation demands certain local and system-specific requirements: Therefore read the chapter "Installation conditions" in your manual for the AAS device thoroughly.

CAUTION

Before moving the device, thoroughly flush the pump and metering hose to prevent reduction agent solution from dripping off. Reduction agent solution is aggressive and attacks clothing.

CAUTION

HS 60 modular has a mass of 14 kg. To prevent health injuries grip the device from the continuous baseplate of the basic module.

5.2 Installation steps for hydride and Hg cold vapor method

ATTENTION

With incomplete installation the device emits a continuous beeping sound. In this case review the installation steps carried out.

5.2.1 Installing the cell unit on the burner block

WARNING

There is a risk of the generation of explosive gas. The cell must be sealed gas-tight for the hydride method (heated operation). Also inspect the polished end faces of the cell. If you notice minor nicks, replace the cell.

ATTENTION

Risk of corrosion! With acid residue remaining in the siphon there is a risk of the cell unit corroding from the effects of the acid vapors. Flush the siphon in the AAS device with 0.5 L water before placing the cell unit onto the mixing chamber connection.

1. Remove the burner head from the burner block.
2. Flush the siphon via the mixing chamber neck with 0.5 L water.
3. Attach the cell unit to the burner block and lock it.
4. ZEEnit 650 P only:
   - Release the attachment screw at the front below the graphite tube furnace, pull the graphite tube furnace out of the sample chamber.
   - Lock the furnace plate with a locking pin.
   - Insert the adapter for the cell unit into the sockets provided on the floor plate of the sample chamber.
   - Place the cell unit onto the adapter and lock it.

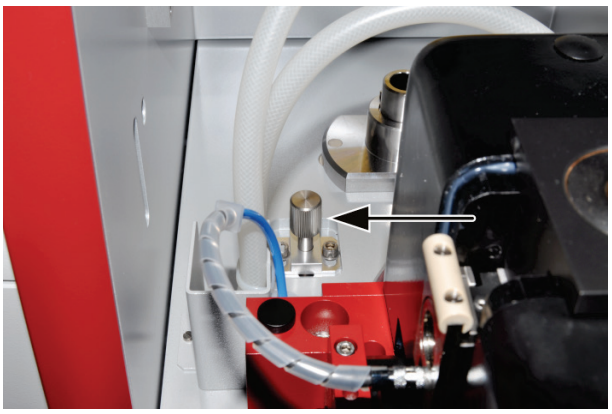
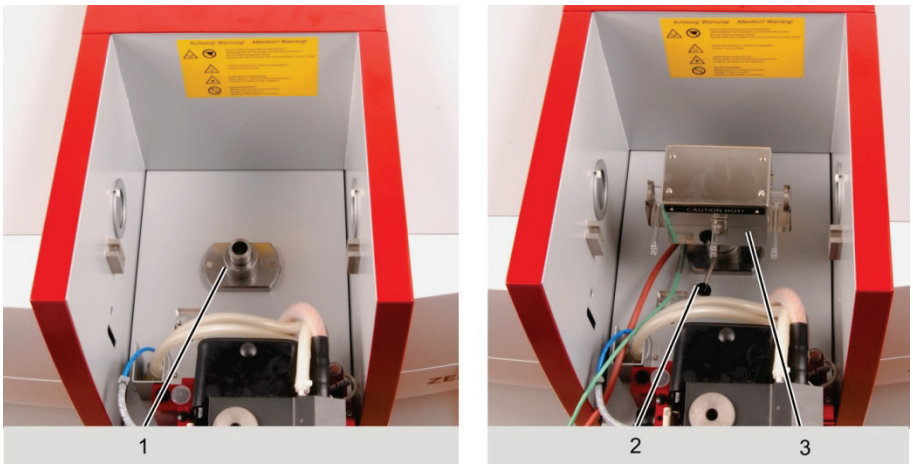


Figure 13 Locking pin at the furnace plate on the ZEEnit 650 P



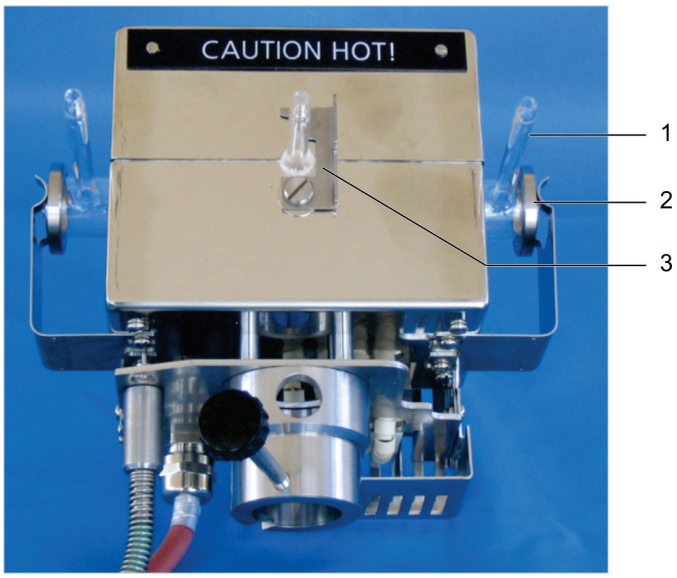
1 cell unit adapter

2 attachment screw

3 cell unit

Figure 14 Adapter and cell unit for Hg/Hydride system on the ZEEnit 650 P

5. Fold the cell unit upwards.



1 quartz cell
2 holder with quartz window
3 lock

Figure 15 Cell unit with quartz cell

6. Insert the cell

Quartz cell for hydride method

Cell for Hg cold vapor method

Figure 16 Cells for hydride and Hg cold vapor methods

For the hydride method:

– Insert the quartz cell, close and lock the cell unit.

– Attach the frame with quartz window on both sides and clamp it in place with the springs. Attach the gas discharge hose to the outer connection and hook the T-piece onto the sample chamber panel at the rear of the sample chamber.

For the Hg cold vapor method:

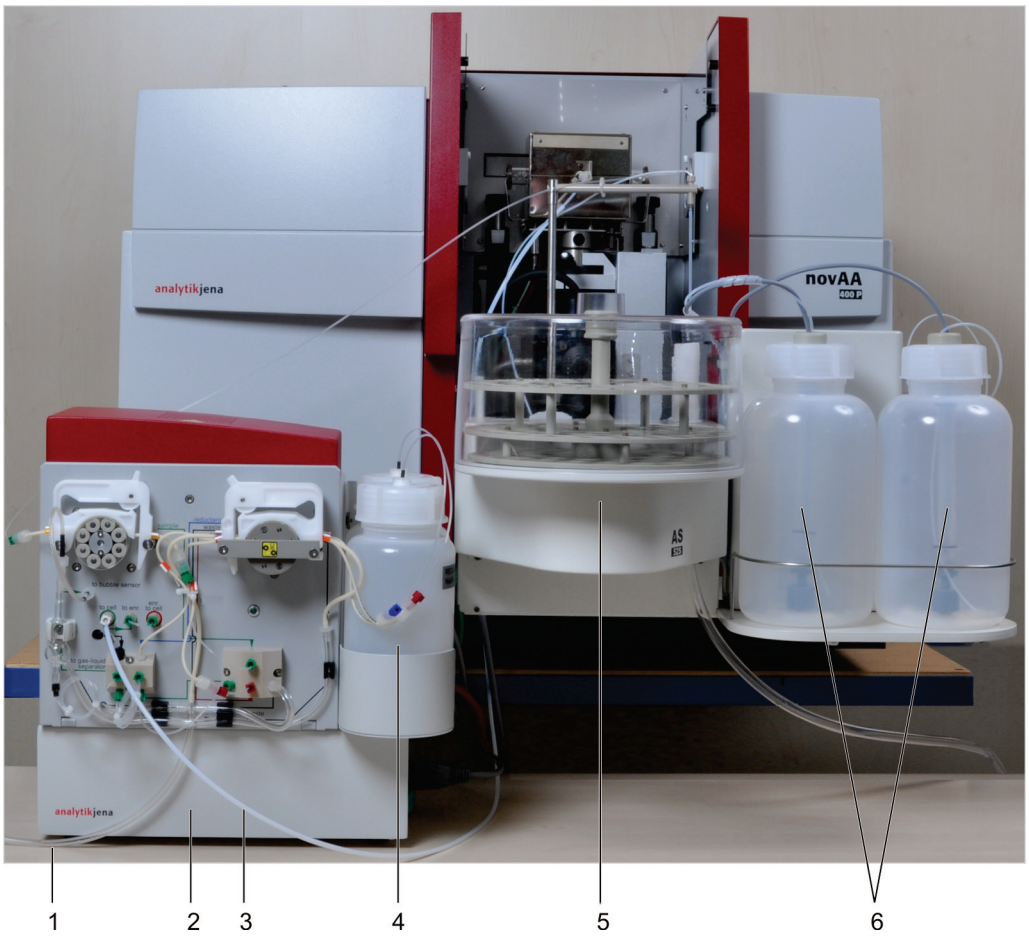
– Insert the Hg cell, close and lock the cell unit.

✓ **This installs the cell unit in the AAS device.**

5.2.2 Installing the HS 60 modular with sampler in the AAS device

IMPORTANT

The voltages (+ 5V/+ 24 V) for the HS 60 modular are provided by the AAS device.



1	drain hose	4	storage bottles for reduction agent, acid
2	basic unit HS 60 modular	5	sampler
3	gas hose to the cell	6	storage bottles for flushing solution, diluent

Figure 17 HS 60 modular with novAA 400 P and sampler

1. Hook the sampler (AS-F/AS-FD) into the sample chamber. Place the HS 60 modular to the right of the AAS device or onto a table next to the AAS device

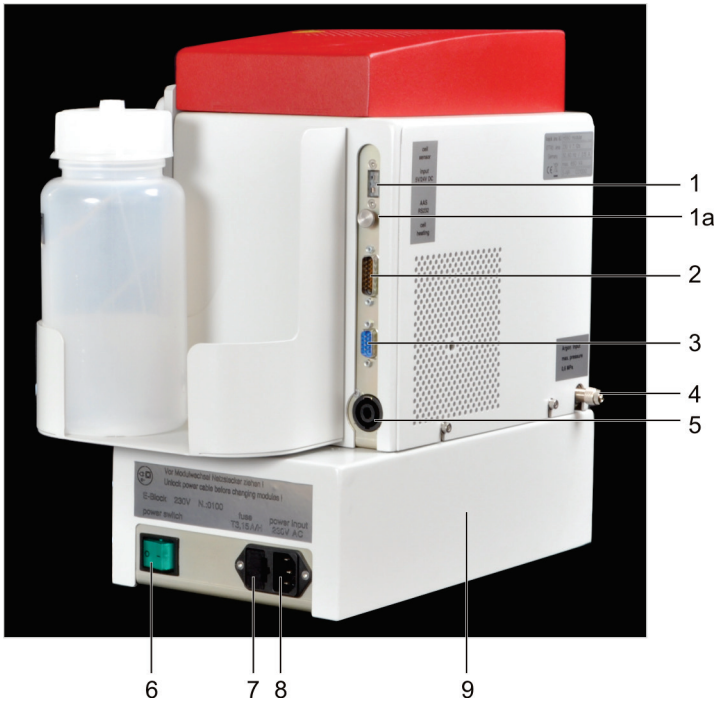
Connect the cell unit:

CAUTION

Voltage may be present at the connection “cell heating”. Observe the safety notices in chapter 3.8.3.

- Heating cable at the connection "cell heating" (5, see Figure 18)
- Temperature sensor cable at the connection "cell sensor" (1)
- Attach the grounding of the sensor cable with knurled screws (1a)

2. Connect the twin cable:
   – Connector "AAS" to jack "AS" of the AAS device
   – D-Sub jack "HS" of the thinner cable to connection "input 5 V/24 V DC" of the HS 60 modular (2)
   – D-Sub jack "AS" of the thicker cable to the sampler connection
3. Connect the signal cable to connector "HS" of the AAS device and to connector "AAS – RS232" of the HS 60 modular (3, see Figure 18).
4. Plug in the mains cable.
5. Connect the Argon hose to the bulkhead fitting on the backplate (4).



1	cell unit temperature sensor connection
1a	grounding knurled screws
2	connection for +5V/+24V supply from the AAS
3	AAS/RS232 interface connection
4	Argon gas connection
5	cell heating connection
6	mains switch
7	fuse drawer
8	mains connection
9	basic module

Figure 18 Hg/Hydride system HS 60 modular – connections on the right-hand side

6. Plug the drain hose onto the free connection of the cross connector. Thread the other end of the hose into the opening in the cover of the waste bottle.
7. Select the hose dryer in accordance with the elements to be detected (for hydride forming elements type "Hy", for mercury type "Hg").
8. Connect the hose dryer to the top outlet of the gas/fluid separator and the connection "to bubble sensor" at the front plate.
9. Connect the reaction gas hose to the cell as follows to the front plate, depending on the cell:

   operating mode "hydride" and "Hg without enrichment"
   – hose to the connection "to cell" (see Figure 19)

   operating mode "Hg with enrichment"
   – hose to the connection "enr. to cell"
   – Close the bridge from the connection "to cell" to the connection "to enr.".
10. Slide the second end of the reaction gas hose onto the center cell connection.

11. Fill the storage bottles with reaction agent and acid.
12. Connect the reduction agent intake hose (with the blue hollow screw) to the reduction agent pump hose (rear hose cartridge) and immerse it up to the stopper into the storage bottle for the reduction agent.
13. Connect the acid intake hose (with the red hollow screw) to the acid pump hose (front hose cartridge) and immerse it up to the stopper into the storage bottle for the acid.
14. Connect the sample intake hose (with the green hollow screw) to the sample pump hose (of the 1-channel pump). Thread it through the slider on the submerged lifting arm and attach it to the thinner cannula.
15. Install the sampler in accordance with the user manual of the AAS device.

    ✓ **The HS 60 modular (with sampler) is installed on the AAS device and is prepared for measurements.**

Switching on sequence

The control board "hydride" of the AAS device is supplied with operating voltages + 5 V/+ 24 V. Mains voltage is only present at the basic module. During the activation initialization the mains frequency is checked.

This leads to the following activation sequence:

1. Switch on HS 60 modular.
2. Switch on AAS device.

    ✓ **First measurements can be started.**

5.2.3 Changing between the operating modes

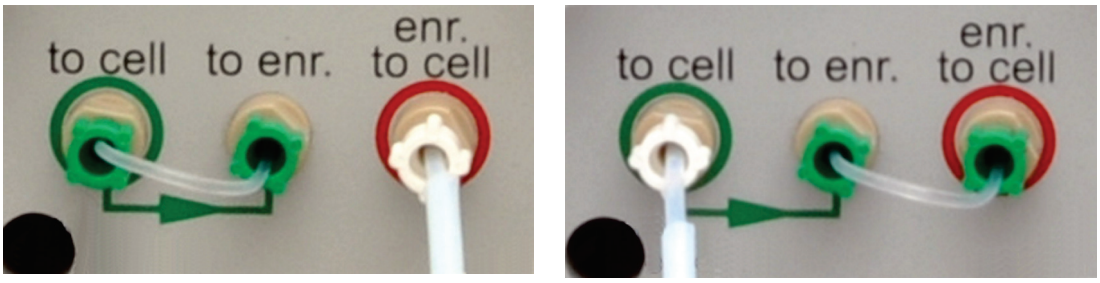
To change between the operating modes "hydride" or "Hg without enrichment" and "Hg with enrichment" the user only needs to change the hose routing at the front plate of the module.

Operating mode "Hg with enrichment"

1. Connect the hose dryer "Hg" between the top outlet of the gas/fluid separator and the connection "to bubble sensor".
2. Close the hose bridge between the connections "to cell" and "to enr." (see Figure 19).
3. Connect the cell hose to the connection "enr. to cell".

Operating modes "hydride" or "Hg without enrichment"

1. Select the hose dryer: "Hy" for the hydride method or "Hg" for the mercury detection.
2. Connect the hose dryer between the top outlet of the gas/fluid separator and the connection "to bubble sensor".
3. Connect the cell hose to the connection "to cell".
4. Close the hose bridge between the outlets "to enr." and "enr. to cell" (or leave it open).



Operating mode "Hg with enrichment" Operating mode "hydride" and "Hg without enrichment"

Figure 19 Hose routing at the front plate in the various operating modes

In addition the corresponding cell must be inserted into the cell unit of the AAS device:

Operating mode "hydride"

– Insert quartz cell and lock with quartz window.

Operating modes "Hg without enrichment" and "Hg with enrichment"

– Insert Hg cell.

5.3 Converting the HS 60 modular

The functional modules flow injection and batch of the Hg/Hydride system are replaceable and can be replaced by the user. In addition, there is an option of retrofitting the device with the "Hg Plus" module.

The software HS Wizard serves as an aid in this process. After starting the program the current device configuration is first queried. Then a target configuration can be chosen. The user is guided through the device conversion.

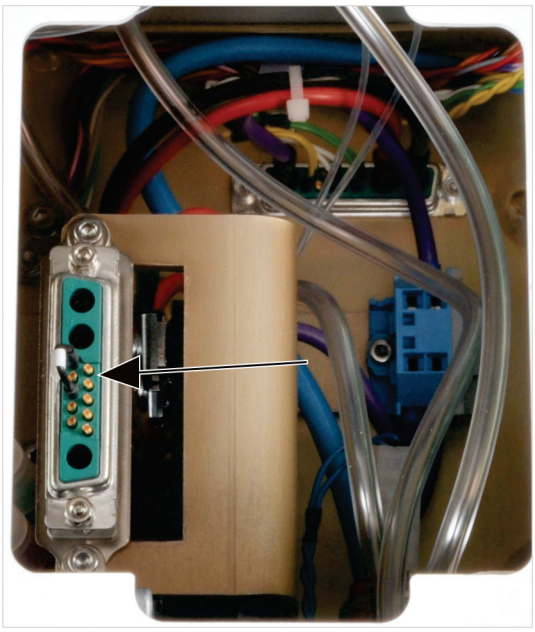
5.3.1 Retrofitting the "Hg Plus" module

WARNING

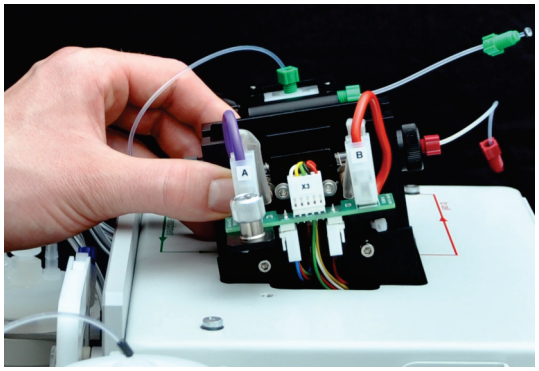
The AAS device and Hg/Hydride system must be switched off for the conversion. There is a risk of electric shock. Disconnect both the mains plug of the Hg/Hydride system and its connections to the AAS device and to the cell unit.

Insert the CD supplied into the PC, start the HS Wizard software and follow the instructions on screen.

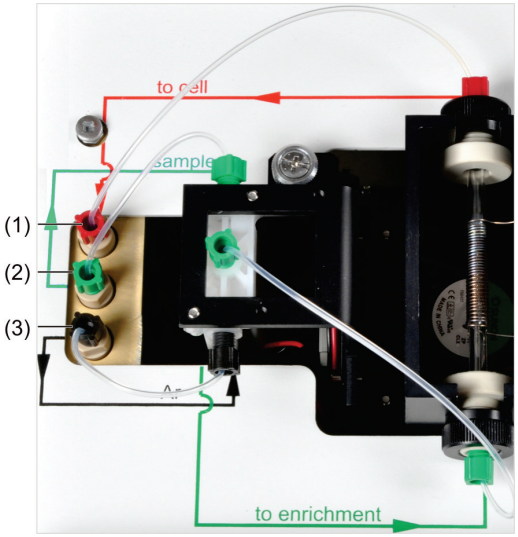
1. Select the spectrometer used.
2. Select the starting configuration of the HS 60 modular.
3. Select the target configuration of the HS 60 modular with enrichment.
4. Retrofitting the "Hg Plus" module:
   – Remove the red hood from the HS 60 modular.



– Pull the shorting plug in the functional module up.



– Thread the "Hg Plus" module, align it with the guide pins and push it down until the plug-in connection has been made.
– Secure the "Hg Plus" module with knurled screws.



Make the hose connections to the flow injection functional module via the frame:

– Hose with the red hollow screws onto the rear connection with the red arrow (1)
– Hose with the green hollow screws onto the center connection with the green arrow (2)
– Hose with the black hollow screws onto the fro0nt connection with the black arrow (3)

– Place the red hood back onto the functional module.
– Connect the system to the mains network, the AAS device and the cell unit. Switch on the device: first HS 60 modular, then AAS. After initialization the devices click on the button [next] in the software.

5. For AAS with serial interface the COM port assigned to the spectrometer must be selected.

✓ **The "Hg Plus" module has been retrofitted and can be tested for operation.**

Functional test of the "Hg Plus" module

1. Start the ASpect LS or ASpect CS software, in the window MAIN SETTINGS select the method HYDRIDE and initialize Available accessories.
2. Exit the Main Settings window with [OK].
3. Click on the button [Hydride syst.].
4. In the tab CONTROL select the following configurations under COLLECTOR:
   – HEATING ON
   ✓ **The heating coil illuminates.**
   – Stop the heating with OFF.
   – COOLING ON
   ✓ **A vertical air flow can be noticed.**
   – Stop the cooling with OFF.
5. Exit the window HYDRIDE SYST.
   ✓ **The "Hg Plus" module is operational.**

5.3.2 Converting the HS 60 modular from the flow injection functional module to the batch module and vice versa

WARNING

The AAS device and Hg/Hydride system must be switched off for the conversion. There is a risk of electric shock. Disconnect both the mains plug of the Hg/Hydride system and its connections to the AAS device and to the cell unit.

After the conversion the Hg/Hydride system may only be put back into operation if the new functional module has been properly screwed to the basic module.

Insert the CD supplied into the PC, start the HS Wizard software and follow the instructions on screen.

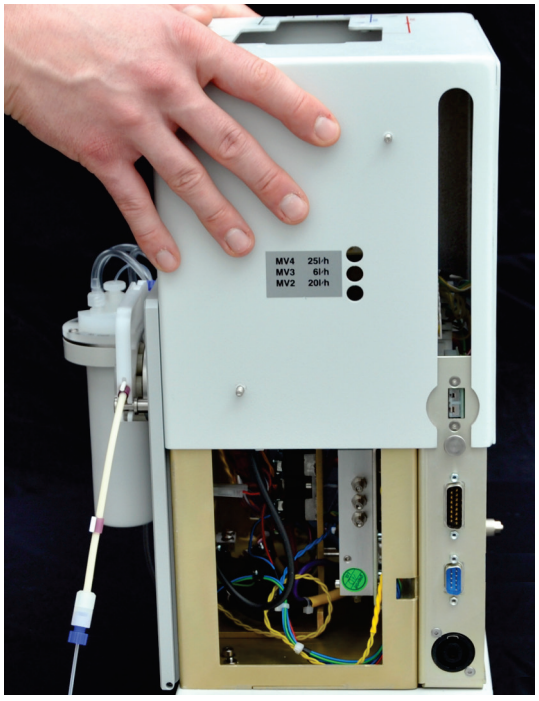
1. Select the spectrometer used in the software.
2. Select the starting and target configuration of the Hg/Hydride system.
3. Remove the red hood.
4. If the Hg/Hydride system has been equipped with a "Hg Plus" module, then this must be removed prior to the conversion:
   – Unscrew the hose connections to the frame.
   – Release the knurled screws at the "Hg Plus" module.
   – Pull the "Hg Plus" module upwards out of the functional module and carefully place it onto the guide pins. Make sure that no hoses etc. are kinked.

5. Converting the functional module:

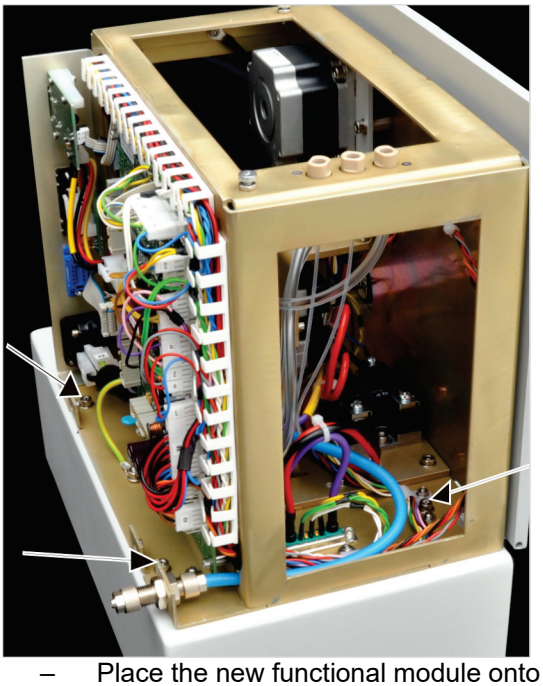
   – Pull the intake hoses out of the storage bottles and wipe them to prevent dripping.

   – Remove the storage bottles from the bottle holder and place them next to the Hg/Hydride system.

   – Releasing the Argon hose.



   – Release the screw fitting at the hood of the functional module and remove the hood.



   – Release the screw fitting of the functional module (4 screws).

   – Pull the functional module upwards out of the basic module and carefully place it onto the guide pins. Please ensure that no hoses are kinked.

   – Place the new functional module onto the basic module, latch it and screw it on.

   – Connect the Argon hose back to the functional module.

   – Place the hood onto the functional module and screw it on.

   – Place the storage bottles into the bottle holder and immerse the intake hoses up to the stopper.

6. Re-installing the mercury module "Hg Plus" (if available) (see 5.3.1):
   - Thread the "Hg Plus" module, align it with the guide pins and push it down until the plug-in connection has been made.
   - Secure the "Hg Plus" module with knurled screws.
   - Make the hose connections to the new functional module via the frame:

     Hose with the red hollow screws onto the rear connection with the red arrow

     Hose with the green hollow screws onto the center connection with the green arrow

     Hose with the black hollow screws onto the fro0nt connection with the black arrow
7. Place the red hood back onto the functional module.
8. Connect the system to the mains network, the AAS device and the cell unit. Switch on the device: first Hg/Hydride system, then AAS. After initialization the devices click on the button [next] in the software.
9. For AAS with serial interface the COM port assigned to the spectrometer must be selected.

   ✓ **The new functional module (batch/flow injection) is now operational.**

5.4 Installation steps for HydrEA method

IMPORTANT

No cell unit is used for the HydrEA method. Instead a shorting plug is plugged onto the temperature sensor connection.

Carry out the installation steps in the following sequence:

1. Install and adjust the sampler graphite (AS-GF) (in accordance with the user manual of the AAS device).
2. Coat the graphite tube.
3. Install the Hg/Hydride system.

5.4.1 Coating the graphite tube with iridium or gold

ATTENTION

The graphite tube must not be coated using the Titanium cannula of the sampler graphite (AS-GF). Otherwise the cannula can no longer be used for measurements. Coat the graphite tube only with the EA configuration of the sampler (i.e. metering hose from MFA).

IMPORTANT

To detect hydride forming elements the graphite tube is coated with iridium. To detect mercury a gold coating is applied.

It is recommended to pipet 50 µL iridium or gold master solution of a concentration of 1 g/L three times with the sampler or by hand into the graphite tube and let it dry.

During the atomization of the introduced substances 150 µg metallic iridium or gold remain adhered to the floor.

The temperatures during coating and bakeout the graphite tube must not exceed 2200 °C or 1000 °C to prevent a loss of iridium or gold.

1. Start the ASpect LS or ASpect CS software, in the window MAIN SETTINGS select the method GRAPHITE FURNACE and the tube type WALL, initialize Available accessories.
2. Exit the window MAIN SETTINGS with [OK].
3. Click on the button [Furnace].
4. Select the tab PLOT and place a tick in the line GRAPHITE TUBE COATING.
5. Define the coating parameters.
   - Cycles = number of pipetting cycles (recommended: 3)
   - Position = position of the master solution on the plate of the sampler
   - Vol. [µL] = sample volume to be pipetted per cycle (recommended: 50 µL)
   - Element = Ir or Au

   The diagram on the screen shows the fixed temperature/time gradient for the tube coating with iridium or gold.
6. Place the sample container with the iridium or gold master solution onto the selected position on the plate of the sampler.
7. Start the coating by clicking on the button [Start].

   ✓ **The graphite tube is coated with iridium or gold.**

Furnace program						
Element	Name	Temp. [°C]	Ramp [°C/s]	Hold [s]	Gas Purge	E/P
Ir	Drying	90	5	40	Max	
	Drying	110	1	40	Max	
	Drying	130	1	40	Max	*
	Pyrolysis	1200	300	26	Stop	
	Atomize	2100	500	8	Stop	
	Clean	2100	0	5	Med	
Au	Drying	80	5	25	Max	
	Drying	90	1	25	Max	
	Drying	110	5	10	Max	*
	Pyrolysis	110	0	6	Stop	
	Atomize	950	500	5	Stop	
	Clean	950	0	5	Med	

5.4.2 Installing HS 60 modular for HydrEA operation

1. At the sampler graphite (AS-GF) release the clamping nut of the hose guide, pull out the metering hose and deposit it in the waste bottle.
2. Push the titanium cannula up to the bend into the hose guide and clamp it on.
3. Arranging the HS 60 modular and the sampler AS-F or AS-FD:
   – If the AAS device has a second sample chamber, the sampler can be hooked into this. The HS 60 modular is placed to the right of the AAS device.
   – If the AAS device does not have a second sample chamber, the HS 60 modular and sampler are either placed on a table before the AAS or to the right or left of it.
4. At the HS 60 modular connect the shorting plug at the connection for the cell unit temperature sensor.
5. Connect the twin cable:
   – Connector "AAS" to jack "AS" of the AAS device
   – D-Sub jack "HS" of the thinner cable to connection "input 5 V/24 V DC" of the HS 60 modular (see Figure 18)
   – D-Sub jack "AS" of the thicker cable to the sampler connection (AS-F/AS-FD)
6. Connect the signal cable to connector "HS" of the AAS device and to connector "AAS – RS232" of the HS 60 modular.
7. Plug in the mains cable.
8. Connect the Argon hose to the bulkhead fitting on the backplate.
9. Do not connect the reaction gas hose and hose dryer. Connect the HydrEA hose with the coupler to the top outlet of the gas/fluid separator and attach the titanium cannula to the sampler graphite (AS-GF).
10. Fill the storage bottle with reduction agent.
11. Connect the reduction agent intake hose (with the blue hollow screw) to the reduction agent pump hose and immerse it up to the stopper into the storage bottle for the reduction agent.
12. Connect the acid intake hose (with the red hollow screw) to the acid pump hose (front hose cartridge) and immerse it up to the stopper into the storage bottle for acid.
13. Connect the sample intake hose (with the green hollow screw) to the sample pump hose (of the 1-channel pump). Thread it through the slider on the submerged lifting arm of sampler AS-F/AS-FD and attach it to the thinner cannula.
14. Install the sampler AS-F/AS-FD according to the manual of the AAS device.

✓ **This installs the HS 60 modular on the AAS device and prepares it for HydrEA operation.**

5.4.3 Adjusting the sampler AS-GF with the titanium cannula

1. Start the ASpect LS or ASpect CS software, in the window MAIN SETTINGS select the method HYDREA and initialize Available accessories.
2. Exit the MAIN SETTINGS window with [OK].
3. Click on the button [Autosampler]. Select the tab TECHN. PARAMETERS and click on the button [Align autosampler to furnace].

   The software now guides you step by step during adjustment in the x,y direction and during the lowering of the titanium cannula.
4. Insert the adjustment aid:
   - novAA 400 P / contrAA 700 / contrAA 600: insert the adjustment aid with the crosshair into the pipetting opening.
   - ZEEnit 700 P / ZEEnit 650 P: remove the left furnace window, remove the graphite tube from the furnace. Insert the adjustment with the hole from the left into the furnace shell.
5. Continue following the software instructions:
   - Align the sampler with adjusting screw 1 in y-direction (sample chamber depth). Secure the adjusting screw with the counter nut.
   - Align the dipping arm in x-direction (parallel to the optical axis) with the adjustment aid using the buttons [left]/ [right]. Fine-align x-direction using adjusting screws 2 and 3 and tighten both screws.
   - Lower the dipping arm until the lower end of the pipetter hose enters the pipetting hole.

   Maximum number of steps: Zeeman furnace – 682 steps, novAA-/contrAA furnace – 566 steps
6. After optimum adjustment save the number of steps in the x direction and the depth in the software by clicking on the button [Next].
   - ✓ **The dipping arm returns to the start position.**
7. Remove the adjustment aid. Preparing the graphite tube:
   - novAA 400 P / contrAA 700 / contraAA 600: Place the graphite insert into the furnace shell.
   - ZEEnit 650 P / ZEEnit 700 P: insert the left furnace window, insert standard graphite tube or coated graphite tube, close the Zeeman furnace.
8. Adjust the the injection depth of the sample in the graphite tube.
   - Loosen the clamping nut, move the pipette hose to the tube bottom. Check the position using the furnace cam.

WARNING

Never use the monitoring mirror with the contrAA! UV radiation is damaging to the eyes.

   - Fasten the pipette hose withe the clamping nut.
9. Set the injection depth to just above the tube bottom (~ 0.5 mm). Save the immersion depth with the button [Finish].
   - ✓ **The sampler has been adjusted and is now prepared for measurements.**

Switching on sequence

The control board "hydride" of the AAS device is supplied with operating voltages + 5 V/+ 24 V. Mains voltage is only present at the basic module. During the activation initialization the mains frequency is checked.

This leads to the following activation sequence:

1. *Switch on* HS 60 modular.
2. Switch on AAS device.
   - ✓ **Sampler, HS 60 modular and AAS device are operational.**

5.4.4 Cleaning the coated graphite tube

IMPORTANT

The iridium- or gold-coated graphite tube can be cleaned in the HydrEA method through bakeout. The iridium coat would evaporate at temperatures above 2200 °C, the gold coating at more than 1000 °C. These temperatures should not be exceeded during cleaning.

1. Start the ASpect LS or ASpect CS software, in the window MAIN SETTINGS select the method HYDREA and initialize Available accessories. Exit the window with [OK].
2. Click on the button [FURNACE]. Select the tab CONTROL and in the area CLEAN FURNACE enter the parameters for cleaning the graphite tube:
   - Temp. [°C] = 2200 (for Ir) or 1000 (for Au)
   - Ramp [°C/s] = 500 (= temperature increase)
   - Hold [s] = 10
3. Start the bakeout of the graphite tube by clicking on the button [Start].
   - ✓ **The graphite tube is cleaned by a brief bakeout. This process can be repeated several times.**

5.4.5 Vaporizing the iridium or gold coat in the graphite tube

IMPORTANT

Once the metal coat has been removed from the graphite tube it can be used as standard graphite tube in the solution analysis or re-used for the HydrEA method.

Before applying a new iridium or gold coat in the graphite tube, the used-up coat must be vaporized with a bakeout temperature of ≥ 2500 °C or ≥ 1800 °C.

1. Start the ASpect LS or ASpect CS software, in the window MAIN SETTINGS select the method HYDREA and initialize Available accessories. Exit the window with [OK].
2. Click on the button [FURNACE]. Select the tab Control and in the area CLEAN FURNACE enter the parameters for cleaning the graphite tube:
   - Temp. [°C] = 2500 (Ir) or 1800 (Au)
   - Ramp [°C/s] = 500 (= temperature increase)
   - Hold [s] = 10
3. Start the vaporization of the metal coat by clicking on the button [START].
   ✓ **The metal coat is removed from the graphite tube through bakeout.**

6 Maintenance and care

6.1 Safety instructions

The user may not perform any service or maintenance work other than that specified in this chapter.

Only the customer service of Analytik Jena or other authorized persons may carry out repairs to the device.

When performing maintenance observe the safety notices listed in chapter "Safety instructions" p. 8.

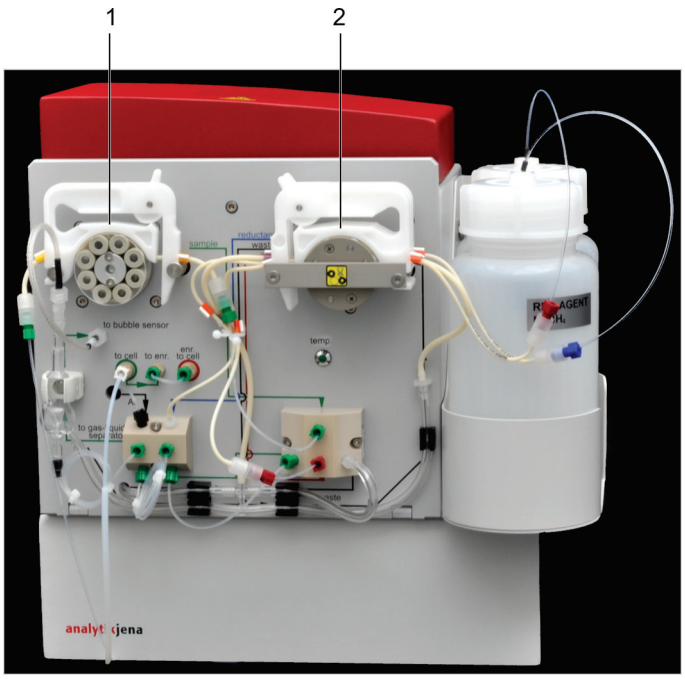
To guarantee sound and safe operation, the HS 60 modular should be inspected annually by the customer service of Analytik Jena.

Only use replacement parts from Analytik Jena. Laboratory parts required for routine operation can be ordered from Analytik Jena.

6.2 Daily maintenance tasks

Tasks for daily commissioning

1. Hook the hose cartridge for the sample into the 1-channel hose pump (sample pump) and that for waste, reduction agent and acid into the 3-channel hose pump (component pump).
2. Pressurize the pump hoses by adjusting the detent lever.
3. Load the sytem with reduction agent and acid:
   - Start the ASpect LS or ASpect CS software, in the window MAIN SETTINGS select the method HYDRIDE and initialize Available accessories. Exit the window with [OK].
   - Click on the button [Hydride syst.]. Select the tab CONTROL and click on the button [Load system].
   - ✓ **The device is operational.**



1 sample pump (1-channel hose pump)
2 component pump (3-channel hose pump)

Figure 20 Hose pumps

Tasks prior to daily shutdown

1. Flush the hoses for sample, reduction agent and acid with distilled water or lightly acidic solution.
2. Pump the hoses empty.
3. Relieve the pump hoses by releasing the hose cartridges.
4. Store the reduction agent solution in the refrigerator.

   ✓ **The device can be switched off.**

6.3 Replacing the fuses

WARNING

There are live components inside the HS 60 modular. There is a risk of electric shock. Prior to performing maintenance always switch off the Hg/Hydride system and disconnect the mains plug.

The mains inlet fuses are located on the right-hand side of the basic module and are labeled. They can be replaced by the user.

Fuse number	Fuse type for mains voltage 230 V	Fuse type for mains voltage 110 V
F1	T3, 15 A/H	T6, 3 A/H
F2	T3, 15 A/H	T6, 3 A/H

6.4 Inspecting and replacing pump hoses

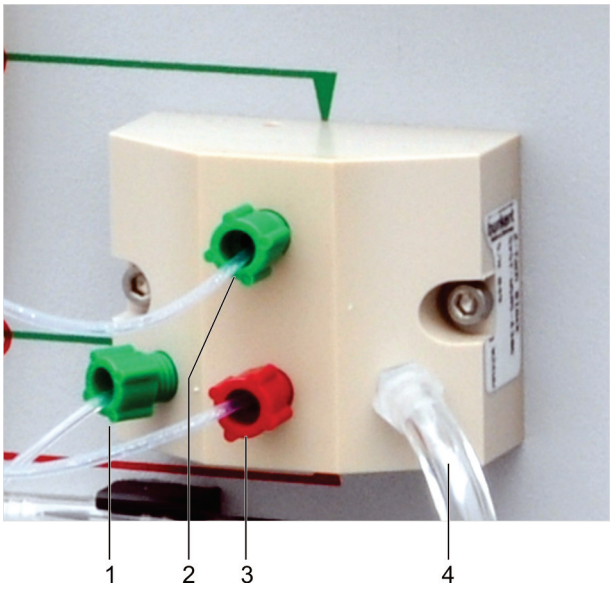
IMPORTANT

Regularly inspect the pump hoses visually for wear and deformation. Always replace the pump hoses for the reduction agent, acid and sample at the same time. This ensures the correct mixing ratio.

CAUTION! Danger of chemical burns!

The solutions used are acidic or alkaline. Before replacing the hoses flush them and pump them empty!

Replacing the sample hose



1 sample to the reactor
2 sample from the sample pump
3 acid from the component pump
4 to waste

Figure 21 2-valve group

In case of deformation in the pump section or irreversible contamination replace the sample hose:

1. Pull the sample hose (MFA) off the cannula of the sampler.
2. Unhook the hose cartridge, remove the sample pump hose (Ismaprene).
3. Detach the sample hose (MFA) at the 2-valve group.
4. Screw the new sample hose into the 2-valve group.
5. Taking note of the pump direction (!) insert the sample pump hose (Ismaprene) into the hose cartridge.
6. Hook in the hose cartridge and press it on.
7. Lead the new sample hose to the sampler and attach it to the intake cannula.

   ✓ **The new sample hose is operational.**

Replacing the pump hoses for the reduction agent and acid

1. Detach the acid pump hose from the 2-valve group.
2. Pull the reduction agent pump hose off the reactor.
3. Pull the corresponding intake hoses off the storage bottles.
4. Unhook the hose cartridges, remove the pump hoses (Ismaprene).
5. Insert the new reduction agent pump hose into the rear hose cartridge, taking note of the pump direction, hook this in and press it on.

   Attach the end of the pump hose to the free connection of the reactor and immerse the intake hose into the storage bottle for the reduction agent.
6. Insert the new acid pump hose into the front hose cartridge, taking note of the pump direction, hook this in and press it on.

   Screw the end of the pump hose into the free opening of the 2-valve group, insert the intake hose into the storage bottle for acid.
7. Move the hose cartridges into the correct locking position.

   ✓ **The new pump hoses are operational.**

6.5 Replacing the hose membrane dryer

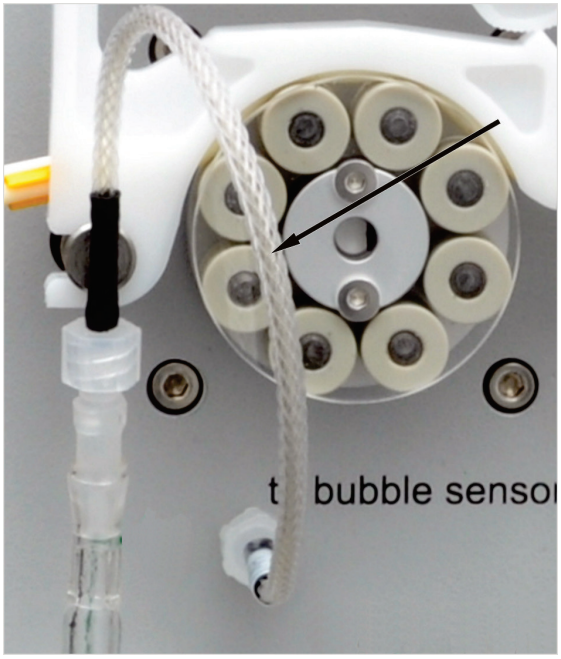


Figure 22 Hose membrane dryer

The hose membrane dryer is functional as long as the surface has not been contaminated with particles or condensate. Always replace contaminated hose membrane dryers. Do not try to clean them.

1. Detach the hose membrane dryer from the coupler at the top connection of the gas/fluid separator and from the connection "to bubble sensor" at the front plate.
2. Screw the new hose membrane dryer to the coupler at the top connection of the gas/fluid separator and to the connection "to bubble sensor".

   ✓ **The new hose membrane dryer is operational.**

6.6 Renewing the hose path

If the hose path from the 2-valve group to the quartz cell is contaminated and a prolonged flushing process with reduction agent solution and acid and gas flushing do not improve the absorption sensitivity, the following hoses must be replaced:

- ❑ Hose from the 2-valve group to the reactor
- ❑ Reactor hose
- ❑ Hose from the reactor to the gas/fluid separator
- ❑ Hose dryer
- ❑ Cell hose (from the front plate to the cell)

1. Unscrew the affected hose or pull it off its connection.
2. Screw in the new hose with a hollow screw or attach it to its connection.

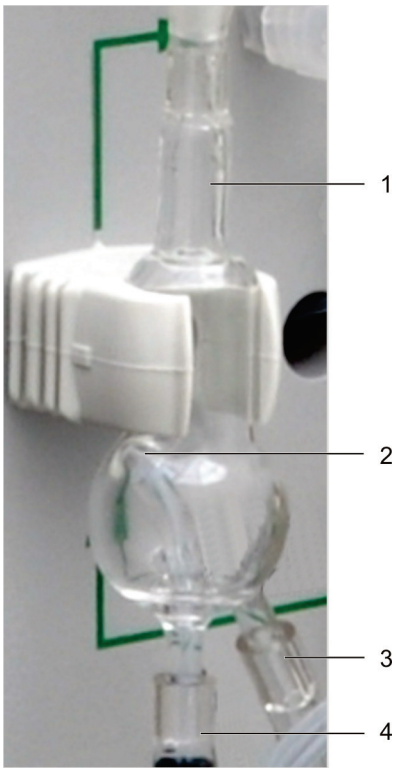
   ✓ **The hose path has been renewed.**

6.7 Cleaning or replacing the gas/fluid separator

First try to remove solid deposits in the gas/fluid separator by cleaning. If this is unsuccessful, replace the separator.

WARNING

The cleaning solution (concentrated hydrochloric acid) is highly corrosive. The vapors irritate the respiratory paths. Wear gloves, protective clothing and safety goggles and work under an extractor!



1 reaction gas outlet
2 bulge
3 fluid waste outlet
4 reaction gas inlet

Figure 23 Gas/fluid separator

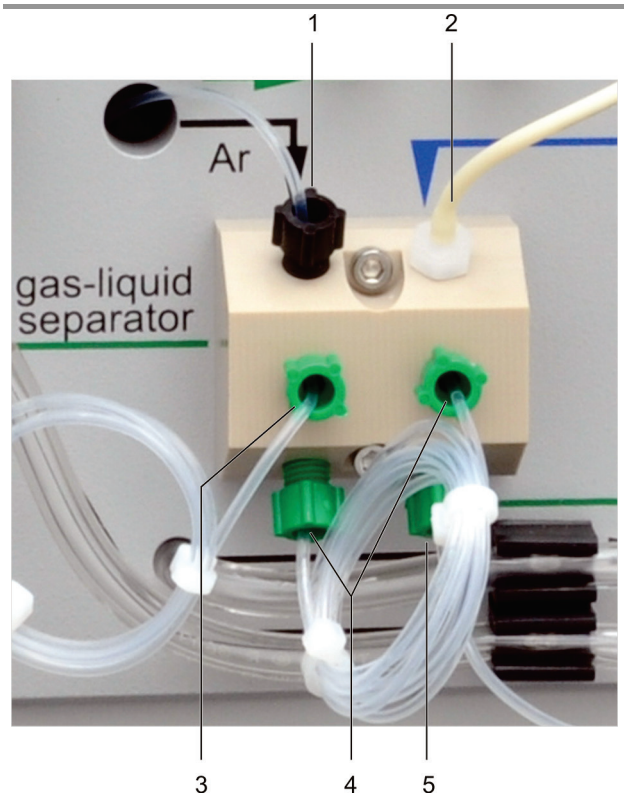
1. Pull the hoses off the gas/fluid separator:
   - Exhaust hose bottom right (3, see Figure 23)
   - Hose from the reactor, bottom (4)
   - Gas outlet hose, top (1)
2. Pull the gas/fluid separator out of the clamp.
3. Clean the gas/fluid separator with concentrated hydrochloric acid (37 %). Allow the acid to act for several hours.
4. Then flush the separator with distilled water.
5. Insert the cleaned or new gas/fluid separator into the clamp.
6. Attach the hoses to the connections of the gas/fluid separator:
   - Exhaust hose bottom right
   - Hose from the reactor, bottom
   - Gas hose onto the outlet connection, top
   - ✓ **The cleaned or new gas/fluid separator is operational.**

6.7.1 Cleaning or replacing the reactor

Clean the reactor if poorly reproducible signals are found, the signals do not arrive at all or a strongly reduced delivery rate is found. If this is unsuccessful, replace the reactor.

WARNING

The cleaning solution (concentrated hydrochloric acid) is highly corrosive. The vapors irritate the respiratory paths. Wear gloves, protective clothing and safety goggles and work under an extractor!



1 Argon inert gas inlet
2 reduction agent inlet
3 outlet of reaction products to the gas/fluid separator
4 hose bridge connections, reactor hose
5 sample inlet (or acid)

Figure 24 Reactor with connections

1. Unscrew or pull off the hoses from the reactor:
   - Pump hose for reduction agent (2, see Figure 24)
   - Sample/acid hose from the 2-valve group (5)
   - Hose bridge connections (4)
   - Gas supply hose (1)
   - Hose to the gas/fluid separator (3)
2. Unscrew the reactor.
3. Dismantle the reactor, unscrew the screw-in connector.
4. Clean the channels in the upper part with cleaning wire.
5. Insert the upper part into concentrated hydrochloric acid (37 %).
6. Clean the Teflon seal.
7. Attach the Teflon seal, securing it with a little adhesive at the corners.

8. First screw on the reactor outside diagonally, then inside, also diagonally.
9. Screw the hose bridge and screw-in connector into the reactor.
10. Screw on the cleaned or new reactor.
11. Screw the hoses into the reactor or attach them to the connections:
    - Pump hose for reduction agent
    - Sample/acid hose from the 2-valve group
    - Hose bridge
    - Gas supply hose
    - Hose to the gas/fluid separator

    ✓ **The cleaned or new reactor is operational.**

6.8 Replacing the gold collector

If the expected sensitivity is not achieved during Hg detection with enrichment, the signals are highly scattered and poorly reproducible, the gold collector must be replaced.

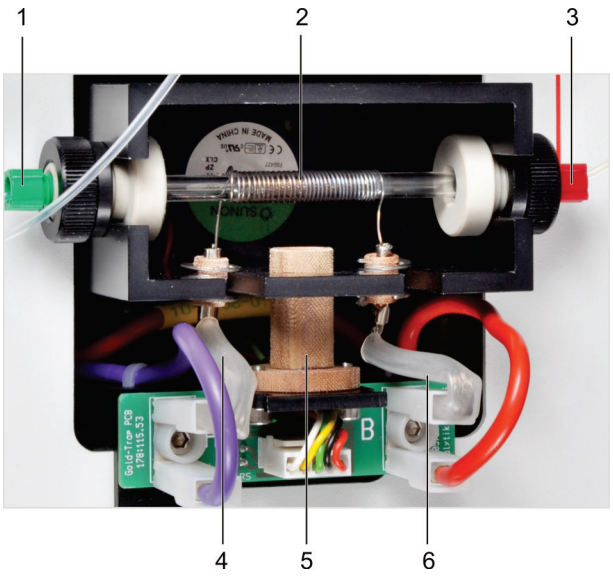
Replacement is also recommended if the gold collector does not bake out completely. This is the case if with large differences in concentrations the new signal level is not reached immediately but only after several measurements.

CAUTION

Risk of burns at the hot collector! Allow the gold collector and heating coil to cool down.

1. Unscrew the MFA hoses from the gold collector.
2. Pull the plug-in contacts of the heating coil (4 and 6, Figure 25) off the circuit board.
3. Release the screw fitting of the gold collector at the compartment, remove the gold collector and heating coil and pull of the screw fitting.
4. Insert the new gold collector into the screw fitting.
5. Insert the gold collector into the compartment whilst simultaneously inserting the isolation sleeves on the heating wire i(2, Figure 25) into the groove.
6. Slide the gold collector up to the stop and screw it tight.
7. Attach the new heating coil with the plug-in contacts to the circuit board.
8. Attach the MFA hoses with the hollow screws to the screw fittings of the collector.

    ✓ **The new gold collector is operational.**



1 supply hose (gas inlet)
2 heating wire
3 discharge hose (gas outlet)
4 heating connection
5 infrared sensor
6 heating connection

Figure 25 Connections at the gold collector

6.9 Cleaning the cell windows and cells

CAUTION

Risk of burns! Before removing the cell windows and before removing the cell allow the cell unit to cool down.

The actual cell temperature is displayed in the software in the window HYDRIDE SYSTEM, tab CONTROL.

Steps when cleaning the cell windows

ATTENTION

Take care not to contaminate the cell windows! Fingerprints burn in. Do not touch the cell windows. Wear rubber gloves!

1. Press the blade spring together and remove the cell window with frame.
2. Clean the cell window with diluted hydrochloric acid.
3. Then rinse the cell window with distilled water and allow it to dry.

✓ **The cell windows have been cleaned.**

Steps when cleaning the cell

WARNING

Hydrofluoric acid is highly corrosive and toxic. Work under an extractor. Wear suitable protective equipment (rubber gloves, rubber apron and face protection).

1. Unlock and unfold the cell unit.
2. Remove the cell and pull off the hoses.
3. Clean the cell 5 to 10 minutes in cold 40 % hydrofluoric acid.
4. Remove the detached film from the inside of the tube with intensive brushing with a suitable round brush under running water.
5. Rinse the cell with distilled water and allow it to dry.

WARNING

Check the end faces of the cell for damage! There is a risk of the generation of explosive gas. Replace damaged cells, do not re-use them!

6. Insert the cell into the heating shell and lock the cell unit.
7. Attach the cell window frame on both sides and clamp it in place with the blade springs. Check the correct contact between the cell window and the cell!

7 Auxiliary and operating materials

WARNING

Protective goggles and protective gloves must be worn when handing auxiliary and operating materials. The safety notices on the labels must be observed.

Sodium borohydride ($NaBH_4$) and sodium hydroxide are strongly corrosive, hygroscopic and, in solution, extremely aggressive. Concentrated hydrochloric acid (HCl, 37 %) is highly corrosive. The standard solution for Arsen (1 g/L) causes severe skin and eye irritation. It is carcinogenic. Care is required during the handling and disposal of these hazardous substances.

The following auxiliary and operating materials are required for the operation of the HS 60 modular:

Auxiliary or operating material	**Preparation**
Reduction agent	
Solution 1: 3.0 % $NaBH_4$ + 1.0 % NaOH (master solution)	Dissolve 7.5 g $NaBH_4$ and 2.5 g NaOH (pellets) in 250 mL dist. water (ultrapure). The solution can be kept 4–6 weeks in the refrigerator.
Solution 2: 0.3 % $NaBH_4$ + 0.1 % NaOH (ready to use)	50 mL of solution 1 is topped up to 500 mL with dist. water. Solution can be kept 1-2 days in the refrigerator.
Acid	
3 % HCl	In a measuring flask 500 mL dist. water is held ready, 70 mL HCl (37 %, smoking) is added and topped up wot 1000 mL with dist. water.
Calibration Arsen	
Flushing solution (for the sampler): approx. 0.2 % HCl	In a measuring flask 500 mL dist. water is held ready, 5 mL HCl (37 %, smoking) is added and topped up wot 1000 mL with dist. water.
Dilution solution (for the sampler): 3.0 % HCl	In a measuring flask 500 mL dist. water is held ready, 70 mL HCl (37 %, smoking) is added and topped up wot 1000 mL with dist. water.
Reduction solution: 5 % KI + 5 % ascorbic acid The solution is used for the reduction of As(+V) to As(+III)	Weigh 2.5 g sodium iodide and 2.5 g ascorbic acid in a clean sealable container and top up with dist. water to 50 mL. The solution can be kept for several days in the refrigerator. Please do not use it any more if it is slightly discolored brown!

Auxiliary or operating material	Preparation
Arsen standard solutions for the hydride method: 0 / 2.0 / 4.0 / 6.0 / 8.0 / 10.0 µg/L As Other standards: 2.0 µg/L: 200 µL solution 2 4.0 µg/L: 400 µL solution 2 6.0 µg/L: 600 µL solution 2 8.0 µg/L: 800 µL solution 2 (preparation see solution 3)	Preparation of the standards via a dilution series Standard example 10 µg/L As Solution 1: 1 g/L As (commercial standard solution) Solution 2: 1 mg/L As To 100 µL of solution 1 7 mL HCl 37 % (p.a.) is added and topped up with dist. water to 100 mL. Solution 3: 10 µg/L As (ready to use) To 1 mL of solution 2 7 mL HCl 37 % (p.A.) and 1 mL reduction solution are added. After waiting 45 min top up with dist. water to 100 mL. Prepare solution 3 fresh every day!
Arsen standard solutions for the HydrEA method: 0 / 0.2 / 0.4 / 0.6 / 0.8 / 1.0 µg/L As Other standards: 0.2 µg/L: 200 µL solution 3 0.4 µg/L: 400 µL solution 3 0.6 µg/L: 600 µL solution 3 0.8 µg/L: 800 µL solution 3 (preparation see solution 4)	Preparation of the standards via a dilution series Standard example 1 µg/L As Solution 1: 1 g/L As (commercial standard solution) Solution 2: 10 mg/L As To 1 mL of solution 1 7 mL HCl 37 % (p.a.) is added and topped up with dist. water to 100 mL. Solution 3: 100 µg/L As To 1 mL of solution 2 7 mL HCl 37 % (p.A.) is added and topped up with dist. water to 100 mL. Solution 3 can be kept 4-5 days. Solution 4: 1.0 µg/L As (ready to use) To 1 mL of solution 3 7 mL HCl 37 % (p.A.) and 1 mL reduction solution are added. After waiting 45 min top up with dist. water to 100 mL. Prepare solution 4 fresh every day!

8 Transport and storage

8.1 Transport

Observe the following notices during transport:

- ❑ The HS 60 modular must always be switched off before transport. Disconnect both the mains plug of the Hg/Hydride system and its connections to the AAS device and to the cell unit.
- ❑ Disconnect the gas supply and detach the Argon hose at the rear of the device.
- ❑ Insufficiently secured components pose a risk of injury! The device components must be secured during transport.
- ❑ Only transport the device in its original packaging! Ensure that all modules are securely screwed on or have been removed and the device is completely empty. Flush the pump and metering hoses thoroughly to prevent reduction agent solution or acid from dripping out. The solutions are aggressive and attack textiles.
- ❑ To prevent health damage the following must be observed when moving the device in the laboratory (lifting and carrying):
  - The Hg/Hydride system has a mass of approx. 14 kg. Since the device does not have handles, grip the device firmly with both hands at the continuous board of the basic module.
  - The guide values and statutory limits for lifting and carrying loads without auxiliary equipment must be observed and adhered to.

8.2 Storage

ATTENTION

Environmental influences and condensate formation can destroy individual components of the HS 60 modular!

The HS 60 modular must only be stored in air conditioned rooms. The atmosphere must be low in dust and free from aggressive vapors.

If the HS 60 modular is not installed immediately after delivery or not required for prolonged periods, it should be stored in its original packaging. A suitable desiccant should be added to the equipment to prevent damage from moisture.

The following storage conditions must be met:

- ❑ Temperature range: -40 °C to +50 °C according to DIN 58390-2
- ❑ max. rel. humidity: max. 90 % at +30 °C

9 Fault removal

Strong foaming can occur in the sample during the hydride and Hg cold vapor methods. In this case a few drops of anti-foaming agent must be added:

Dow-Corning DB 110A, silicon anti-foaming agent or Octanol

For unknown samples the foaming must be tested. In case of foaming disconnect the the hose membrane dryer at the outlet of the gas/fluid separator.

If foam is carried along during too strong a reaction, the measuring process must be stopped immediately. The Hg/Hydride system should be switched off.

10 Disposal

At the end of its service life the HS 60 modular and all its electronic components must be disposed of in accordance with the applicable regulations as electronic waste.