

Operating Manual

HS 55 modular

Hg/Hydride System Batch Mode

HydrEA System Batch Mode



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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 User manual notes

The HS 55 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 55 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 55 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 55 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 3 "Safety instructions" p. 8 and in the description of the relevant work steps.

2 Technical data

Technical data	
Designation/type	HS 55 modular
Techniques (dependent on the configuration installed)	Hydride method Hg cold vapor method without enrichment Hg cold vapor method with enrichment HydrEA method
Operating modes	Discontinuous (batch mode)
detectable elements	As, Bi, Hg, Sb, Se, Sn, Te
Dimensions (W × H × D)	360 × 370 × 270 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: 1.0 % NaBH ₄ + 0.3 % NaOH Tin(II) chloride SnCl ₂ as alternative for Hg detection
Main functional groups	
1-channel hose pump for reduction agent transport	Equipment: Ismaprene hose ID = 2.06 mm; plug: purple Pumping speed: 4 stages
Reaction unit	Batch module: PTFE cup with conical floor
"Hg Plus" module	Gold collector: 0.5 g gold/platinum alloy AuPt 10 as fine mesh Bakeout temperature: 630 °C controlled Cooling: axial fan
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: purge gas: F2 = 15 L/h, transp. gas: F3 = 6 L/h, F4 = 25 L/h, F3+F4 = 31 L/h

Operation times	Wash time 1: Time for purging the reaction cup of air AZ wait time: Waiting time immediately prior to zero balance. Pump time: time during which the 1-channel pump pumps reduction agent into the cup. Wash time 2,3: times for the transport of the reaction gas with the Argon flow. 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
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
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 55 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 55 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) may result.



CAUTION

Indicates a potentially hazardous situation which, unless avoided, may cause 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 on the HS 55 modular

Safety symbols have been attached to the HS 55 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 55 modular and accessories:



General warning



Warning against hot surface



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

3.5 Technical condition

The HS 55 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 55 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 55 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 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 55 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 hose to prevent reduction agent solution from dripping out. The solution is aggressive and attacks 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 55 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 or repair of the device.

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 55 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 55 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 55 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 always 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 55 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 55 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 55 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 handling 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 applicable regulations for disposal of these materials must be meticulously observed.
- When measuring cyanide-containing material, ensure that prussic acid is not contained 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:

- During operation, do not remove the reaction beaker during the reaction and measuring times.
- Always ensure that the cells with the windows are closed and gastight. 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 only carry out the tasks listed in the chapter "Service 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. 38 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 blocks (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 NaBH₄. 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 NaBH₄ tin(II) chloride SnCl₂ 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 55 modular

The HS 55 modular consists of the basic module, the batch 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 55 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

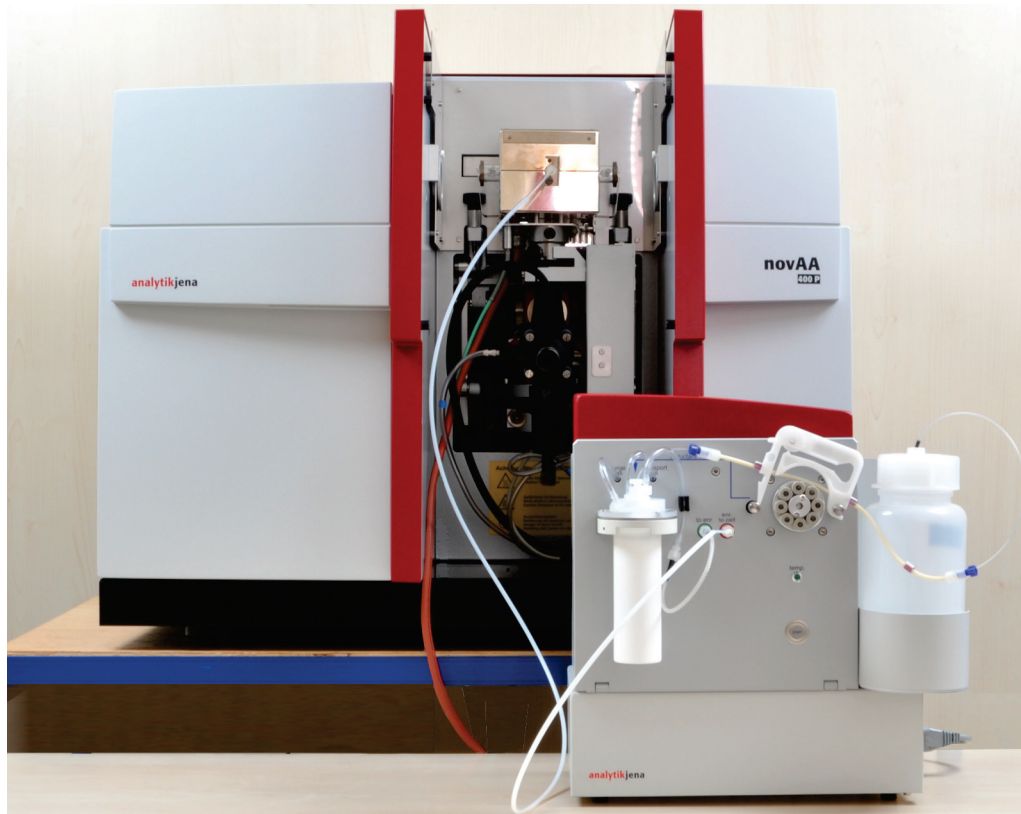


Figure 1 HS 55 modular with AAS novAA 400 P

At the front plate of the batch functional module all functionally relevant assemblies are located:

- 1-channel hose pump for the reduction agent transport
- Batch module as reaction unit
- Push button to start the measuring process

The pump hose is also located here. It is easily accessible and can be replaced by the user. The colored line on the front plate identifies the hose routing and thereby facilitates maintenance.



- 1 Batch module
- 2 1-channel hose pump
- 3 "Start" push button
- 4 Basic module

Figure 2 HS 55 modular (front view)

On the inside of the functional module the 4-valve group for the gas supply is located. On the right-hand side of the device is the storage bottle for the reduction agent in a holder. Here the electrical connections are located.

The "Hg plus" module is inserted from the top into the batch 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 55 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 is pipetted into the reaction cup (max. 20 mL), this is clamped gas-tight to the head of the batch module. The 1-channel hose pump transports the reduction agent into the reaction cup. The fast and partly vigorous reaction releases gaseous metal hydride or atomic Hg vapor. These are transported by the Argon flow and also released hydrogen either directly into the quartz cell or for Hg enrichment through a gold collector. The enriched Hg is released during the bakeout of the gold collector and transported to the Hg cell by a directly connected Argon flow.

After the measurement the reaction cup is removed, flushed and filled with new sample.

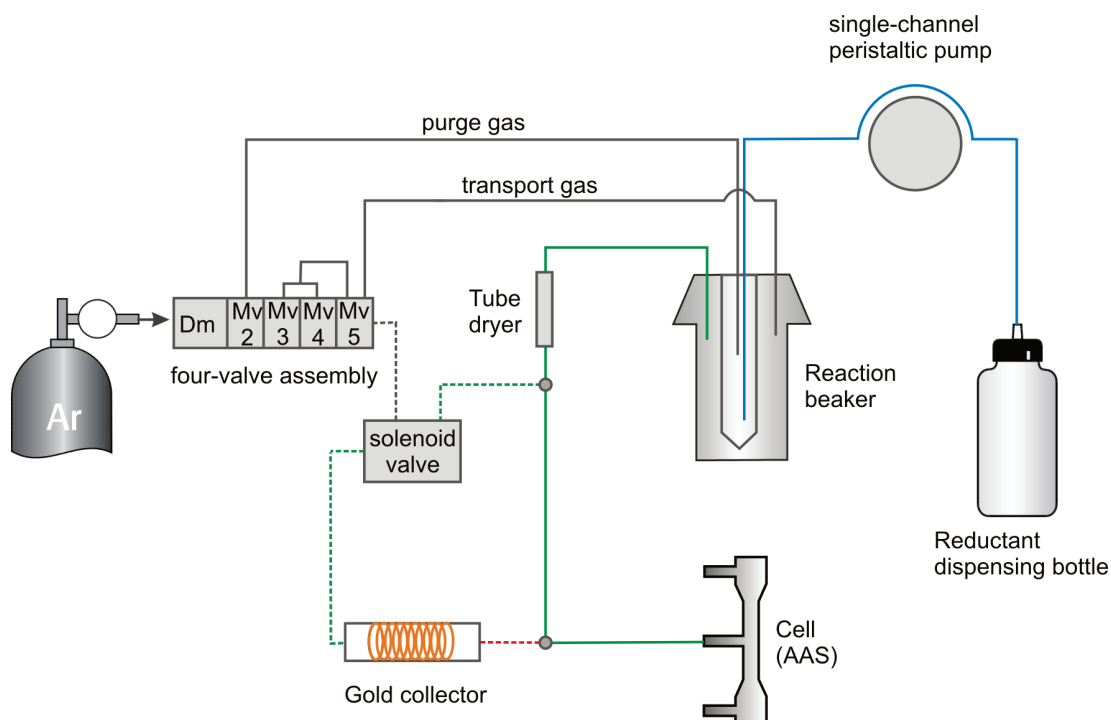


Figure 3 Functional diagram of the HS 55 modular



IMPORTANT

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

4.3 Design of the HS 55 modular

4.3.1 1-channel hose pump

The 1-channel hose pump is equipped with an adjustable snap-in cartridge and an Isma-prene hose with 2.06 mm inside diameter. It only runs during the pump time and transports reduction agent in 4 selectable speed stages.

4.3.2 Batch module

The batch module consists of a reaction cup with conical floor for sample volumes of 1–20 mL and the head.

The head contains:

- Supply connections for purge gas (15 L/h) and transport gas 6 L/h + 25 L/h for Argon
- Gas outlet to the cell "to cell")
- Flange seal for reaction cup
- Metering tip

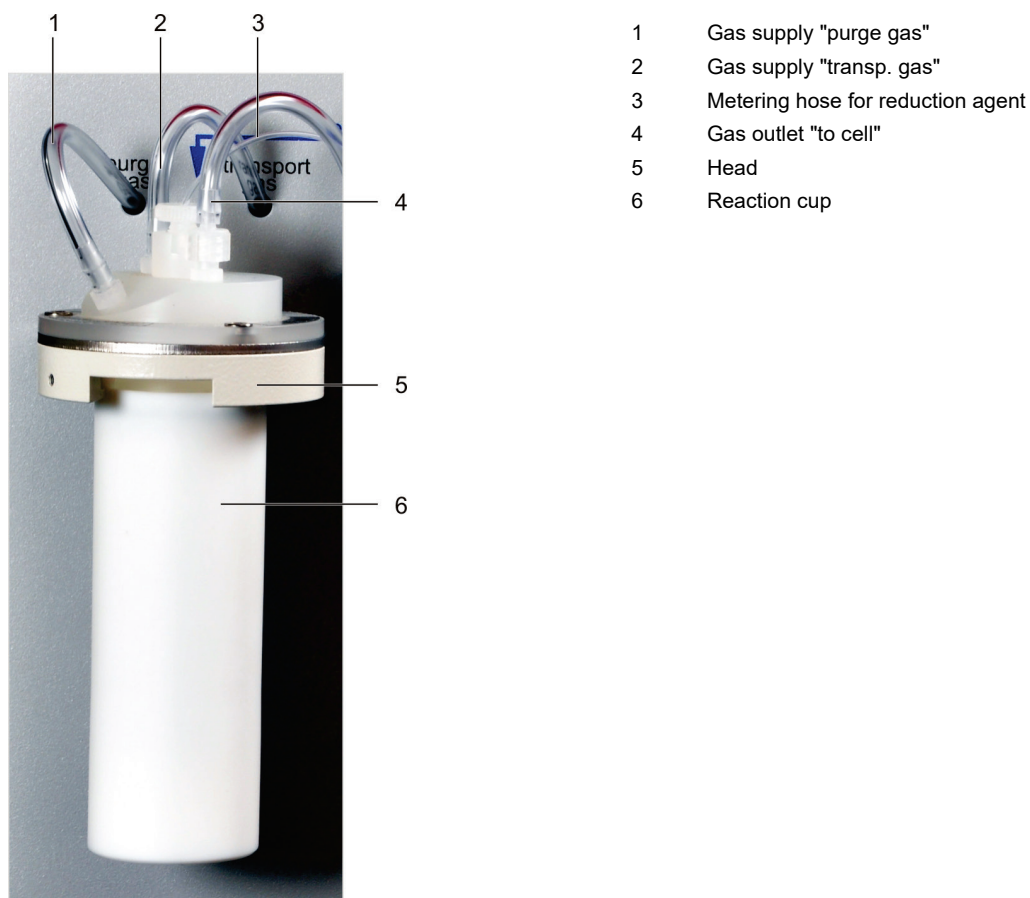


Figure 4 Batch module

The reduction agent and purge gas are transported through the metering tip to the floor of the reaction cup. The reaction with the sample starts from below and is accelerated through the released reaction gas and the purge gas. The purge gas drives the released metal hydride or Hg vapor out of the sample solution. The transport gas enters the reaction cup from above and transports the metal hydride or Hg vapor from the reaction cup to the cell or the gold collector.

4.3.3 4-valve group for gas control

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

Valve MV2: F2 with 15 L/h as constant "purge gas" through the pipetting tip of the batch module

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

4.3.4 "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. It comprises in addition to the compartment with the gold collector, sensor and fan a 3/2-way solenoid valve. 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.

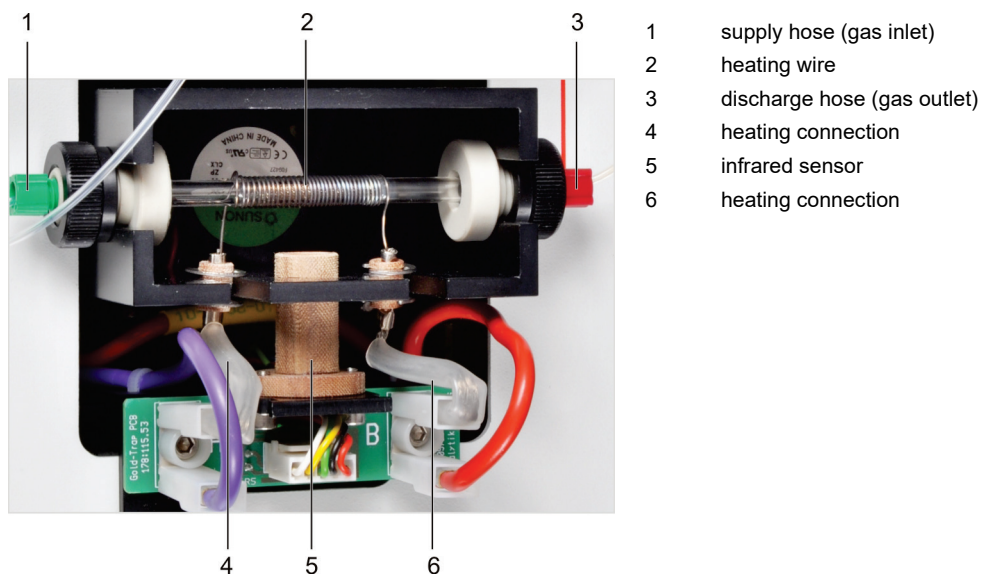


Figure 5 gold collector

4.4 Measuring processes



IMPORTANT

Measurements can be started either via the HS 55 modular or the software of the AAS device. Information about performing the measurements can be found in the separate user manual of the AAS device.

4.4.1 Discontinuous operation without enrichment

The measuring process is started immediately after preparing the reaction cup with new sample. During the flushing time 1 (Prewash time) the reaction cup is flushed of air with maximum Argon flow. Flushing time 1 is omitted for Hg to prevent the highly volatile element from being driven out too early.

During the AZ waiting time (AZ wait time) constant measuring conditions are established in the cell for the selected gas flow for the measuring phase, then the zero value (AZ) is determined over 4 s as fixed time. For Hg the AZ wait time has been blocked, the AZ time has been reduced to 2 s.

The measuring start and pump start are simultaneous. During the pump time the 1-channel pump transports reduction agent into the reaction cup. The flushing time 2 extends the selected gas state. During flushing time 3 (Wash time 3) the reaction cup and cell are flushed of reaction products with maximum gas flow.

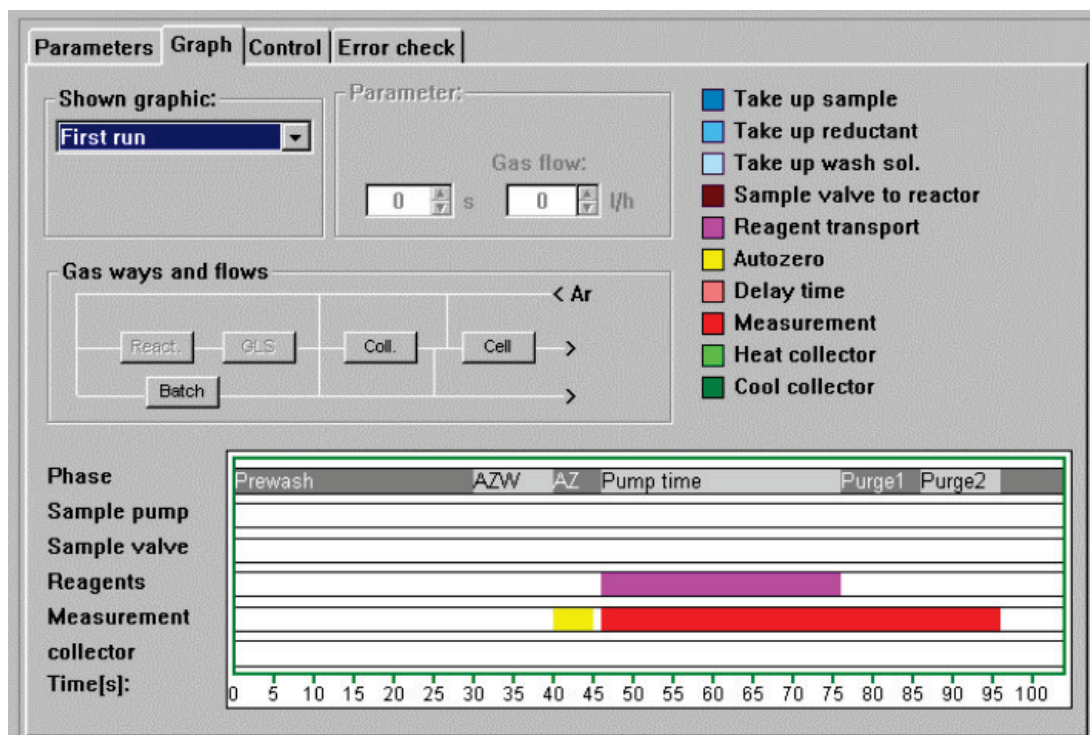


Figure 6 Measuring process for mercury; discontinuous operation

4.4.2 Discontinuous operation with Hg enrichment

The measuring process can be started after preparing the reaction cup with new sample. Flushing of the reaction cup is omitted.

During the pump time the 1-channel pump transports reduction agent into the reaction cup. The release Hg vapor is transported during the pump time and the subsequent flushing time 1 (Wash time 1) to the gold collector where it is enriched.

During the fixed AZ waiting time (AZ wait time) of 2 s fresh gas is transported with a freely selectable flow rate directly via the gold collector to the cell and creates constant conditions for the zero value measurement (AZ) over 4 s.

The bakeout of the gold collector, the flushing time 2 and the measurement are started at the same time. The selected gas flow transports the Hg released in the gold collector to the cell.

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

During the subsequent flushing time 3 the gold collector and cell are flushed clear with maximum gas flow.

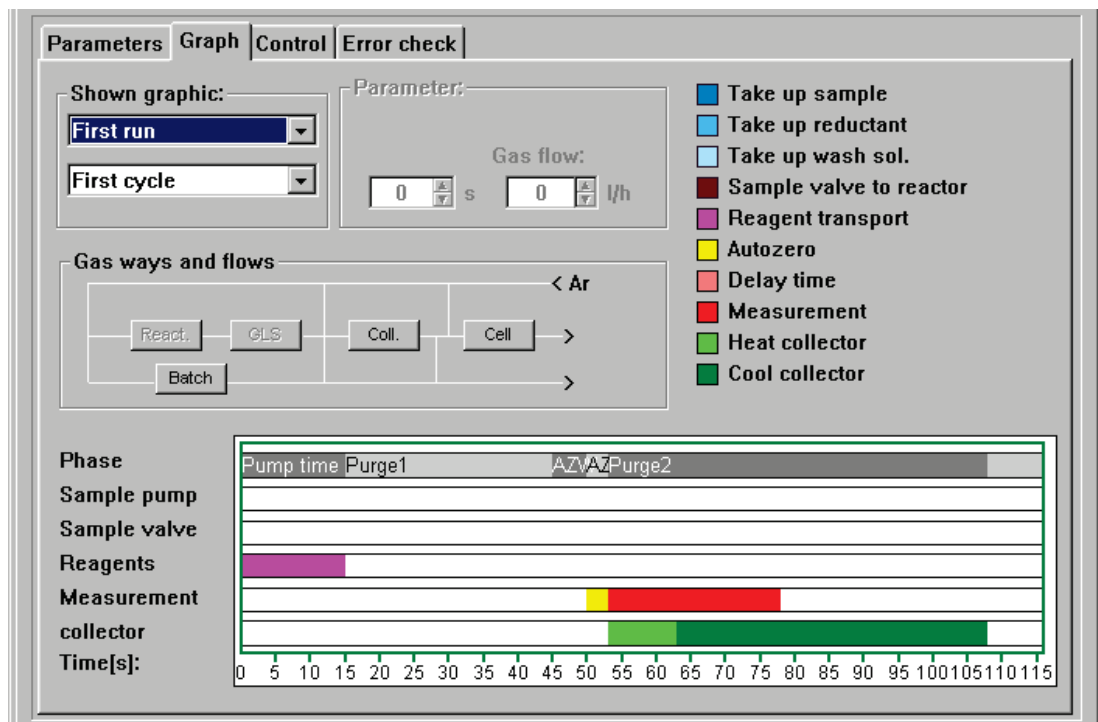


Figure 7 Measuring process for mercury; discontinuous operation with enrichment

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 55 modular has a mass of 14 kg. To prevent health injuries it is gripped 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.

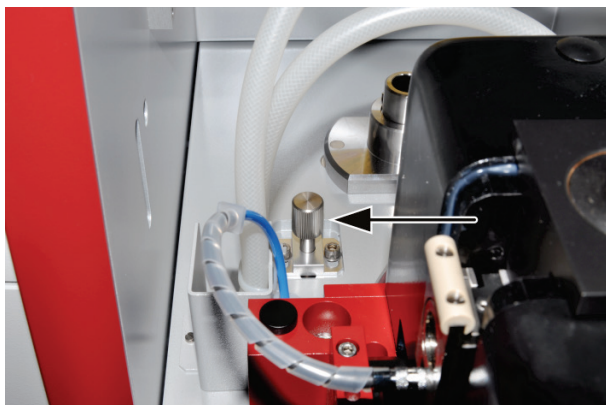


Figure 8 Locking pin at the furnace plate on the ZEEnit 650 P

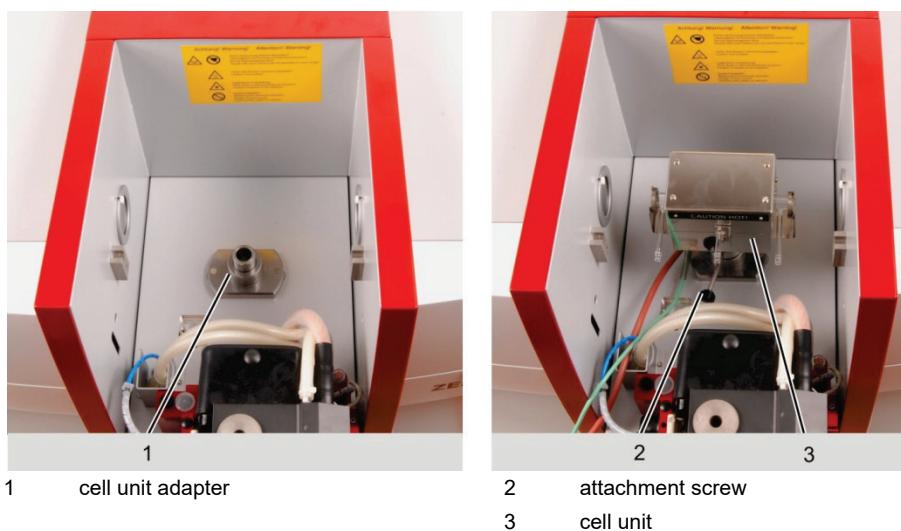


Figure 9 Adapter and cell unit for Hg/Hydride system on the ZEEnit 650 P

5. Fold the cell unit upwards and insert the cell.



Quartz cell for hydride method



Hg cell

Figure 10 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.**

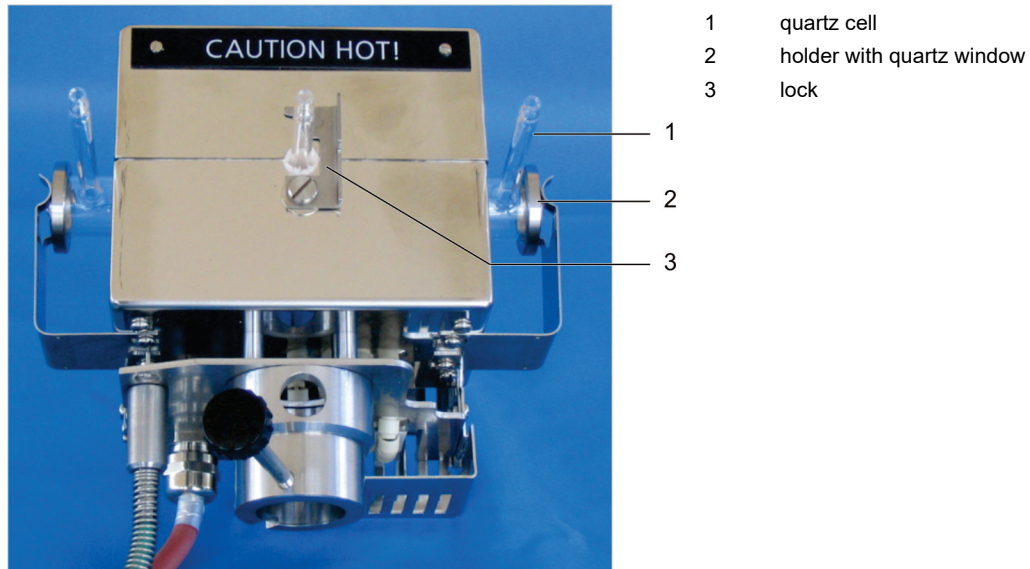


Figure 11 Cell unit with quartz cell (for the hydride method)

5.2.2 Installing the HS 55 modular on the AAS device



IMPORTANT

The voltages (+ 5V/+ 24 V) for the HS 55 modular are provided by the AAS device.

1. *Place the* HS 55 modular to the right of the AAS device or onto a table next to the AAS

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 12)
- 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 55 modular (2) The D-Sub jack "AS" remains empty.

- Connect the signal cable to connector "HS" of the AAS device and to connector "AAS – RS232" of the HS 55 modular (3).

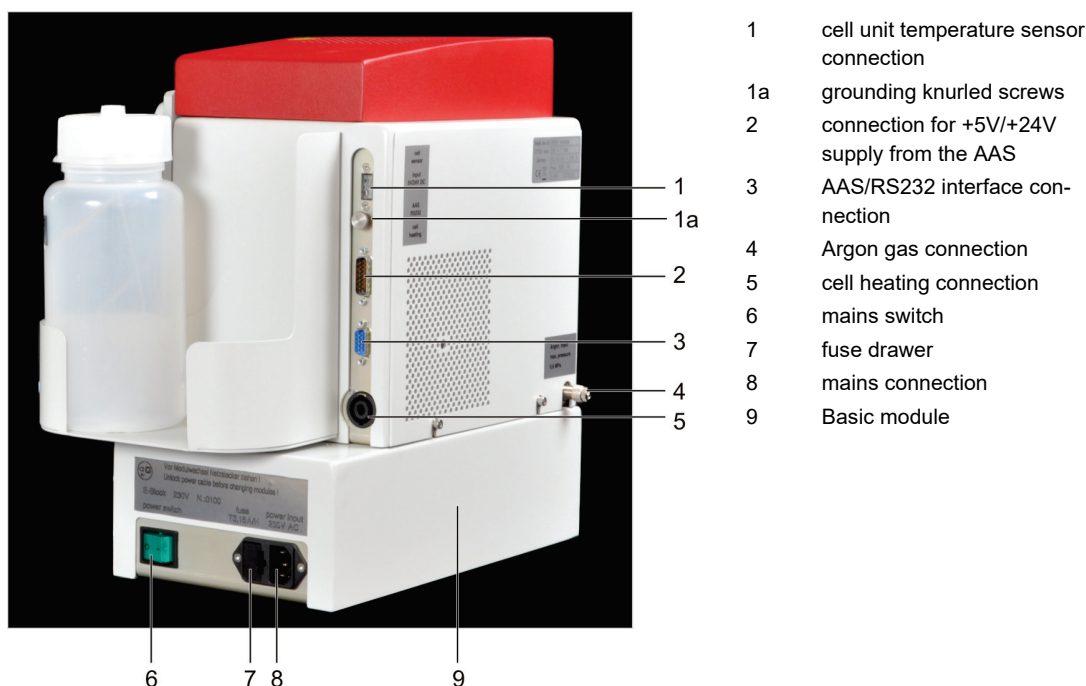


Figure 12 Hg/Hydride system HS 55 modular – connections on the right-hand side

- Plug in the mains cable.
- Connect the Argon hose to the bulkhead fitting on the backplate (4).
- Select the drying hose in accordance with the elements to be detected (for hydride forming elements type "Hy", for mercury type "Hg") and connect it.
 - Screw the connection hose "Batch module drying hose" onto the "to cell" outlet of the batch module.
 - Connect the drying hose to the outlet.
- Connect the drying hose and cell hose to each other or to the batch module:

For the operating modes "Hg without enrichment" or "Hydride":

 - Connect the drying hose "Hg" or "Hy" via the female Luer coupling to the cell hose.

For the operating mode "Hg with enrichment":

 - Screw the drying hose "Hg" to the connection "to enr." (to enrichment) at the front plate, screw the cell hose to the connection "enr. to cell" (see Figure 13).
- Fill the storage bottle with reduction agent (1.0 % NaBH_4 / 0.3 % NaOH).
- Connect the reduction agent intake hose (with the blue hollow screw) to the pump hose of the 1-channel pump and immerse it up to the stopper into the storage bottle for the reduction agent.
- Hook in the hose cartridge at the HS 55 modular and adjust the locking lever.
 - ✓ **This installs the HS 55 modular on the AAS device and prepares it 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 the HS 55 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 batch module.

Operating modes "hydride" or "Hg without enrichment"

1. Select the drying hose: "Hy" for the hydride method or "Hg" for the mercury detection.
2. Connect the cell hose to the drying hose via the female Luer coupling.
3. Connect the drying hose via the connection hose to the gas outlet "to cell".

Operating mode "Hg with enrichment"

1. Connect the drying hose "Hg" via the connection hose to the gas outlet "to cell" at the batch module and the connection "to enr." at the front plate.
2. Connect the cell hose to the connection "enr. to cell" at the front plate.



Operating mode "Hg without enrichment"



Operating mode "Hg with enrichment"

Figure 13 Hose routing at the front plate of the batch module in the various operating modes

In addition the 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 55 modular

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

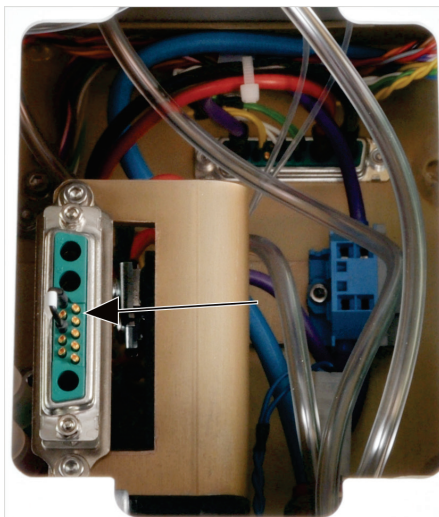


WARNING

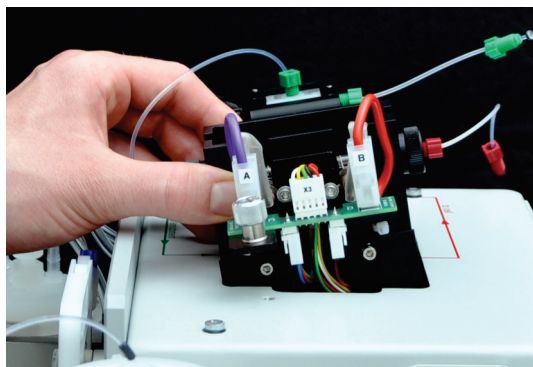
Risk of electric shock! Do not touch! The AAS device and Hg/Hydride system must be switched off for the conversion. Disconnect both the mains plug of the Hg/Hydride system and its connections to the AAS device and to the cell unit.

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

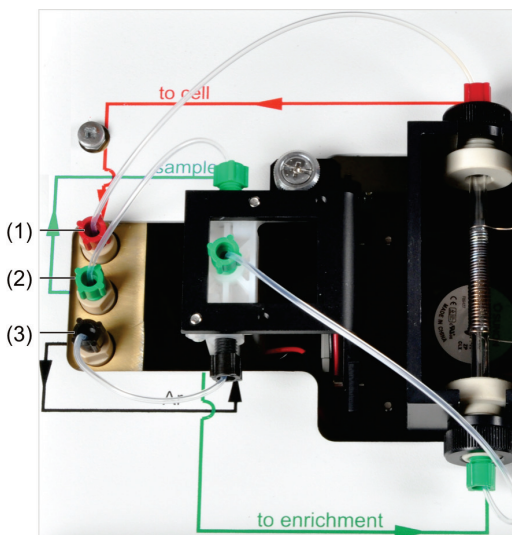
1. Select the spectrometer used.
2. Select the starting configuration of the HS 55 modular.
3. Select the target configuration of the HS 55 modular with enrichment.
4. Retrofitting the "Hg plus" module:
 - Remove the red hood from the HS 55 modular.



- 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 batch 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 front 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 55 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 55 modular from the batch functional module to the flow injection module and vice versa



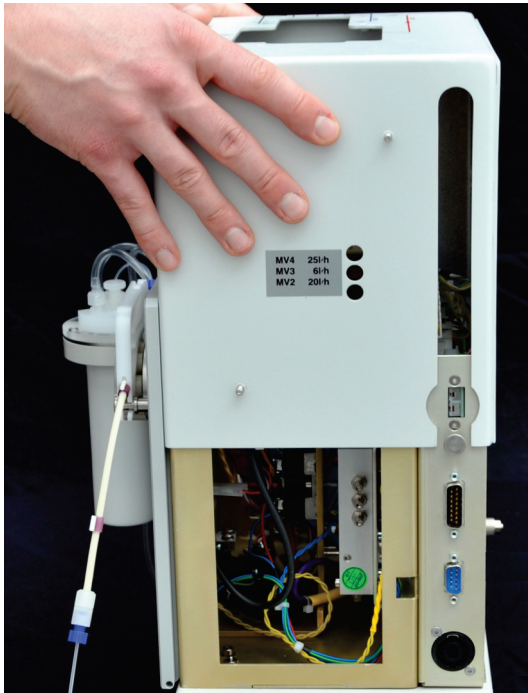
WARNING

Risk of electric shock! Do not touch! The AAS device and Hg/Hydride system must be switched off for the conversion. 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 hose out of the storage bottle and wipe it to prevent dripping.
 - Remove the storage bottle from the bottle holder and place it next to the Hg/Hydride system.
 - Detach the Argon hose from the bulkhead fitting on the backplate.



- 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 bulkhead fitting on the rear of the device.
 - Place the hood onto the functional module and screw it on.
 - Place the storage bottle into the bottle holder and immerse the intake hose up to the stopper.
6. Re-installing the 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 (see chapter 5.3.1):

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 front 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 (flow injection/batch) 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 autosampler AS-GF. Otherwise the cannula can no longer be used for measurements. Coat the graphite tube only with the EA configuration of the AS-GF (MFA metering hose).



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 graphite or by hand into the graphite tube and let it dry. During the atomization of the introduced substances 150 µg metallic iridium or gold then 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. [$^{\circ}\text{C}$]	Ramp [$^{\circ}\text{C}/\text{s}$]	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 55 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. *Arrange the* HS 55 modular either on a table in front of the AAS device or to its right/left.
4. 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 55 modular (see Figure 12) The D-Sub jack "AS" remains empty.
6. Connect the signal cable to connector "HS" of the AAS device and to connector "AAS – RS232" of the HS 55 modular.
7. Plug in the mains cable.
8. Connect the Argon hose to the bulkhead fitting on the backplate.
9. Connect the HydrEA hose with the coupler to the connector "to cell" of the batch module and attach it to the titanium cannula of the sampler.
10. Fill the storage bottle with reduction agent (1.0 % NaBH₄ / 0.3 % NaOH).
11. Connect the reduction agent intake hose (with the blue hollow screw) to the pump hose of the 1-channel pump and immerse it up to the stopper into the storage bottle for the reduction agent.
12. Hook in the hose cartridge at the HS 55 modular and adjust the locking lever.
 - ✓ **This installs the HS 55 modular on the AAS device and prepares it for the HydrEA method.**

5.4.3 Adjusting the sampler graphite 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 window MAIN SETTINGS 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 cross-hair 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 autosampler 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 / contrAA 600: Place the graphite insert into the furnace shell.
 - ZEE nit 650 P / ZEE nit 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 with 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 autosampler 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 *the* HS 55 modular.
2. Switch on AAS device.
 - ✓ **Sampler, HS 55 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 coat at more than 1000 °C. These temperatures should not be exceeded.

3. 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].
4. 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
5. 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 3 "Safety instructions".

To guarantee sound and safe operation, the HS 55 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 into the 1-channel pump.
 2. Pressurize the pump hose by adjusting the detent lever.
 3. Load the system with reduction agent:
 - 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 place a tick in the line COMPONENTS PUMP.
 - Start the pump and let it run until reduction agent drips into the reaction cup held under the metering tip.
- ✓ **The device is operational.**

Tasks prior to daily shutdown

1. Flush the reduction agent hose with distilled water or a weak acidic solution.
 2. Pump the hose empty.
 3. Relieve the pump hose by releasing the hose cartridge at the 1-channel pump.
 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 55 modular. Prior to performing maintenance always switch off the HS 55 modular 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 Servicing the reduction agent hoses



IMPORTANT

Regularly inspect the pump and metering hose for the reduction agent visually for wear and deformation. The hoses need not only be replaced in case of heavy wear but also if the reduction agent (NaBH_4 / SnCl_2) is replaced.



CAUTION! Danger of chemical burns!

The reduction agent solution is corrosive. Before replacing the hose flush it and pump it empty!

Replacing the complete hose set

With excessive wear or contamination the complete hose set for the reduction agent (intake hose, pump hose, metering hose) must be replaced.

1. Release the clamping screw at the batch module and pull out the metering hose (MFA).
2. Unhook the hose cartridge, remove the pump hose.
3. Insert the new pump hose into the cartridge. Note the pump direction!
4. Hook in the hose cartridge and press it on.
5. Feed the new metering hose (MFA) into the batch module. Tighten the clamping screw firmly.



IMPORTANT

The hose end must be approx. 10 mm above the pipette tip.

6. Insert the intake hose up to the stopper into the storage bottle.

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

6.5 Replacing the drying hose

The drying hoses are functional as long as the surface has not been contaminated with particles or condensate. Always replace contaminated drying hoses.

1. Detach the drying hose from the hose section at the outlet "to cell" of the batch module and from the cell hose or the inlet "to enr." at the front plate.
2. Connect the new drying hose to the hose section at the outlet "to cell" of the batch module and the cell hose.

In the operating mode "Hg with enrichment" tighten the second hose end with the hollow screw to the connection "to enr." at the front plate.

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

6.6 Inspecting and replacing the flange seal in the batch module

After an extended operating period the sealing ring at the flange can lose its elasticity and sealing effect and must then be replaced.

1. Pull off the reaction cup from the flange with one turn.
2. Visually inspect the sealing ring on the flange, remove worn sealing ring and replace by new sealing ring.
3. Slide the reaction cup onto the flange and lock with one turn.

✓ **The flange seal has been replaced.**

6.7 Replacing the gold collector

If the expected sensitivity is not achieved during Hg detection with enrichment, the absorption peaks 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 14) 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 14) 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.**

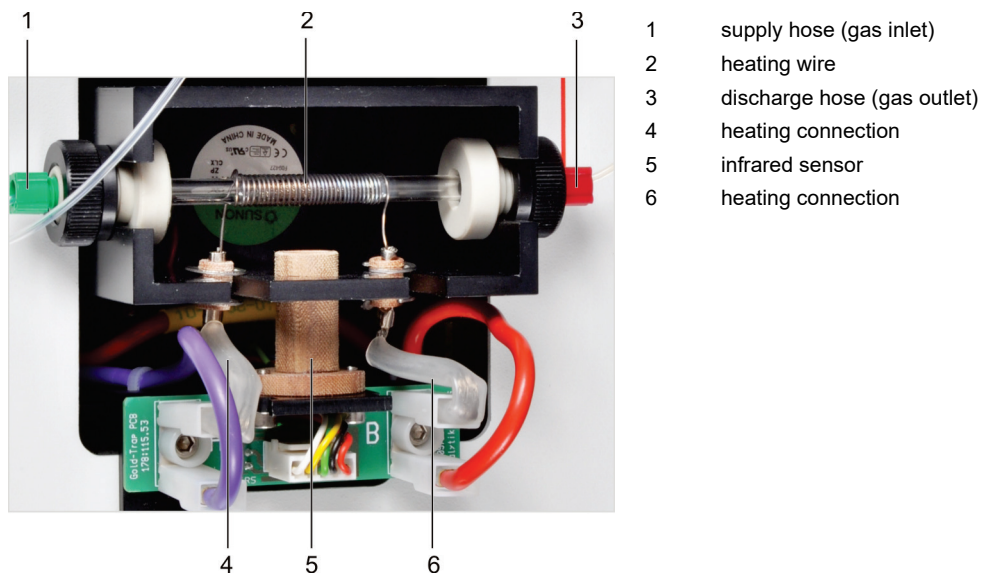


Figure 14 Connections at the gold collector

6.8 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 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 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! Danger of explosive gas forming! Replace damaged cell and do not re-use it!

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!
 - ✓ **The cleaned cell is operational again.**

7

Auxiliary and operating materials

**WARNING**

Protective goggles and protective gloves must be worn when handling auxiliary and operating materials. The notes on the labels have to 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 55 modular:

Auxiliary or operating material	Preparation
Reduction agent	
Solution 1: 3 % NaBH_4 + 1 % 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: 1.0 % NaBH_4 + 0.3 % NaOH (ready to use)	100 mL of solution 1 is topped up to 300 mL with dist. water. Solution 2 can be kept 1-2 weeks in the refrigerator.
Calibration Arsen	
Reduction solution: 5 % KI + 5 % ascorbic acid	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 is used for the reduction of $\text{As}(+\text{V})$ to $\text{As}(+\text{III})$	The solution can be kept for several days in the refrigerator. Please do not use it any more if it is slightly discolored brown!
Arsen standard solutions for the hydride method: 0 / 2.0 / 4.0 / 6.0 / 8.0 / 10.0 $\mu\text{g/L As}$	Preparation of the standards via a dilution series
	Standard example 10 $\mu\text{g/L As}$ 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. 100 mL with dist. water.
Other standards: 2.0 $\mu\text{g/L}$: 200 μL solution 2 4.0 $\mu\text{g/L}$: 400 μL solution 2 6.0 $\mu\text{g/L}$: 600 μL solution 2 8.0 $\mu\text{g/L}$: 800 μL solution 2 (preparation see solution 3)	Solution 3: 10 $\mu\text{g/L As}$ (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. up to 100 mL with distilled water. Prepare solution 3 fresh every day!

Auxiliary or operating material	Preparation
<p>Arsen standard solutions for the HydrEA method: 0 / 0.2 / 0.4 / 0.6 / 0.8 / 1.0 µg/L As</p> <p>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)</p>	<p>Preparation of the standards via a dilution series</p> <p>Standard example 1 µg/L As Solution 1: 1 g/L As (commercial standard solution)</p> <p>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. 100 mL with dist. water.</p> <p>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. 100 mL with dist. water. Solution 3 can be kept 4-5 days.</p> <p>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. 100 mL with dist. water. Prepare solution 4 fresh every day!</p>

8 Transport and storage

8.1 Transport

Observe the following notices during transport:

- The HS 55 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.
- 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. Thoroughly flush the pump and metering hose to prevent reduction agent solution from dripping out. The solution is aggressive and attacks 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.

8.2 Storage



ATTENTION

Environmental influences and condensate formation can destroy individual components of the HS 55 modular!

The HS 55 modular must only be stored in air conditioned rooms. The atmosphere must be low in dust and free from aggressive vapors.

If the HS 55 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, silicion anti-foaming agent or Octanol.

For unknown samples the foaming must be tested. If foam is carried along to the quartz cell, the measuring process must be stopped immediately.

10 Disposal

At the end of its service life the HS 55 modular and all its electronic components must be disposed of in accordance with the applicable regulations as electronic waste.