analytikjena
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Operating Manual

HS 55 (Batch Mode)
Hg/Hydride System
HydrEA System

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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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Technical Documentation

Analytik Jena GmbH+Co. KG

Table of contents

1 Basic information

1.1 About this operating manual

Content

This operating manual describes the HS 55 Hg/hydride system.

The device is intended to be operated by qualified specialist personnel observing the operating manual.

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

Conventions

Instructions for actions occurring in chronological order are numbered and combined into action units.

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

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

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

Symbols and signal words used in this manual

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

WARNING

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

CAUTION

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

NOTICE

Provides information on potential material or environmental damage.

1.2 Intended use

The Hg/hydride system must only be used in connection with an atomic absorption spectrometer by Analytik Jena.

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

2 Security

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

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

2.1 Safety labeling on the device

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

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

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

Warning symbol	Meaning	Comment
	Warning against a hazard location	On the cell heating connection: dangerous electrical voltage warning. Only connect and disconnect the connection cable when the device is switched off.
	Warning about hot surface	On the red device hood: there is a risk of burns on the Hg Plus Upgrade Modul (optional). Allow the module to cool down before opening the hood.
"Caution Hot" warning sign	Warning about hot surface	On the cell heating: risk of burns at the cell heating. Keep a safety distance during operation.

Mandatory action labels/information symbols	Meaning	Comment
	Disconnect the power plug prior to assembly or disassembly and opening of the device.	On the power switch/power input: Switch off the device and disconnect the power plug from the power connection prior to assembly or disassembly and opening of the device.
	For People's Republic of China only	The device contains controlled substances. Analytik Jena GmbH+Co. KG warrants that these substances will not be released from the device within the next 25 years provided the device is employed as intended.

2.2 Requirements for the operating personnel

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

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

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

2.3 Safety instructions – transport and installation

Incorrect installation can create serious hazards. This may cause an electric shock.

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

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

- The device has no carrying handles. To transport the device, grip the device firmly at the bottom with both hands and lift it.
- Ensure that the device is completely empty. Flush the pump and metering hoses thoroughly to prevent reduction agent solution from dripping out. The solution is corrosive and harmful to health and damages clothing.
- Remove the storage bottle for reduction agent. Empty the bottle before transport.
- Insufficiently secured components pose a risk of injury. During transport, secure the device components as specified in this operating manual.
- Risk of damage to health due to improper decontamination! Perform a professional and documented decontamination of the device before returning it to Analytik Jena. The decontamination report is available from Service when registering the return. Without a completed decontamination report, the acceptance of the device will be refused. The sender may be liable for damage caused by inadequate decontamination of the device.

2.4 Safety instructions – operation

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

Observe the following:

- The device may only be operated if all items of protective equipment (e.g. covers in front of electronic components) are in place, properly installed and fully operational.
- The sound condition of the protection and safety equipment must be checked regularly. Any defects must be corrected as soon as they occur.

- Protective and safety equipment must never be removed, modified or switched off during operation.
- Free access to the power switch on the right 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 ventilation grilles or slots etc. may cause the device to break down or may cause damage to it.
- The cell unit becomes very hot during operation. Hot components must not be touched during or directly after the operation of the device. The cooling times to room temperature (at least 1 hour) must be observed.
- Keep flammable materials away from the cell unit.
- The gold collector (Hg Plus Upgrade Modul, optional) also becomes very hot during operation. Only open the red device hood when the module has cooled down.
- Caution when handling glass components. Risk of broken glass and therefore risk of injury!
- During operation, there is a risk of crushing at the hose pump. Long hair and baggy clothing can become caught in the pump and drawn in. Wear suitable hair protections and tight-fitting clothing.

2.4.1 Safety instructions – protection against explosion and fire

The device may not be operated in an explosive environment.

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

2.4.2 Safety instructions – electrical equipment

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

- The power plug must be connected to a proper power outlet to ensure that the device meets protection class I (ground connector). The device may only be connected to power sources whose nominal voltage is the same as that on the rating plate of the equipment. Do not replace the removable power cable of the device with a power cable that does not meet the specifications (with no protective ground conductor). Extensions of the supply cable are not permitted!
- The basic module and the system components may only be connected to the mains when they are switched off.
- Electrical connection cables between the basic module and the system components may only be connected or disconnected when the device is switched off.
- Before opening the device, the device must be switched off via the main switch and the power plug must be disconnected from the power outlet!
- Work on the electronics may only be carried out by the customer service of Analytik Jena and specially authorized technicians.

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

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

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

- Only operate the device when the exhaust unit is active. Ensure good room ventilation in working rooms.
- Cleaning with hydrofluoric acid must be carried out in an exhaust chamber. The operator must wear a rubber apron, gloves and a face mask when handling hydrofluoric acid.
- When measuring cyanide-containing material, ensure that prussic acid cannot be generated in the waste bottle.

Protective goggles and rubber gloves have to be worn when handing reagents.

- Sodium borohydride ($NaBH_4$) and sodium hydroxide (NaOH) are strongly corrosive, hygroscopic and, in solution, extremely aggressive.

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 beaker to the cell outlet must be kept oxygen-free. To this end take the following measures:

- Always ensure that the cell with the windows is closed and gastight. Replace the cell even in the case of small cracks or damage to the front sides.
- Guide the gas from the cell outlet to the exhaust unit.

Observe the following:

- The operator is responsible for carrying out suitable decontamination should the device become contaminated externally or internally with dangerous substances.
- Splashes, drops or larger liquid spillages should be removed using an absorbent material such as cotton wool, laboratory wipes or cellulose.
- For biological contamination, wipe the affected area with a suitable disinfectant, such as an Incidin Plus solution. Then wipe the cleaned areas so that they are dry.

- The only suitable cleaning method for the housing is wipe disinfection. If the disinfectant has a spray nozzle, apply disinfectant to a suitable cloth before using it on the device.
  Work particularly carefully and cleanly with infectious material because the device cannot be decontaminated as a whole.
- Before using a cleaning or decontamination procedure other than that prescribed by the manufacturer, the user is required to check with the manufacturer that the intended procedure will not damage the device. Safety labels attached to the device must not have methanol applied.

2.5 Safety instructions – maintenance and repair

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

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

- Only clean the exterior of the device with a slightly moistened, non-dripping cloth. Use only water and, if required, customary surfactants.
- All maintenance and repair work on the device must only be carried out when the device is switched off (unless specified otherwise).
- The gas supply must be shut off before performing any maintenance or repair work (unless specified otherwise).
- Allow the device to cool down before any maintenance work or replacement of system components.
- Use only original spare parts, wear parts and consumables. They have been tested and ensure safe operation. Glass part are wear parts and are not subject to the warranty.
- All protective equipment must be reinstalled and checked for proper function when the maintenance or repair work is complete.

2.6 Behavior during emergencies

- If there is no immediate risk of injury, switch off the device and the connected system components immediately in hazardous situations or in the event of an accident and/or disconnect the power plugs from the power outlets.
- Close the gas supply as soon as possible after switching off the devices.

3 Function and design

3.1 Function and measuring principle

Hydride method

The hydride method enables the matrix-free detection of the elements As; Bi; Sb; Se; Sn; Te. It is based on the formation of gaseous metal hydrides through reduction of the acidic samples with sodium borohydride ($NaBH_4$). The carrier gas and the released hydrogen transport the metal hydrides to the quartz cell. There they gradually decompose through collisions with gas particles and the glass wall at temperatures of 850 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.

Cold vapor method

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

HydrEA method

The HydrEA method combines the hydride or Hg cold vapor method with the graphite tube method. It is used for the highly sensitive selective detection of the hydride-forming elements As; Bi; Sb; Se; Sn; Te or Hg with the graphite tube furnace.

The Hg/hydride system generates the gaseous metal hydrides or the atomic Hg vapor, respectively. The autosampler for the graphite tube furnace (AS-GF) transfers the analytes with the carrier gas argon into the graphite tube furnace. There the metal hydrides are enriched on the iridium-coated standard tube for wall atomization at a preheating temperature of 300 °C. They decompose in this process. At a temperature of 2100 °C, the deposited metal atoms atomize again. The generated atom vapor cloud absorbs the primary radiation on the resonance line.

The atomic Hg vapor is enriched on the gold-coated standard tube at 65 °C. At a temperature of 950 °C, the Hg atoms atomize again.

Overview of Hg/hydride systems

Hg/hydride system	Function
HS 50	Simplest batch system with pneumatic operating principle The quartz cell is heated by the acetylene-air flame.
HS 55	Batch system with electrically heated cell unit The user fills the sample solution manually into the reaction beaker of the Hg/hydride system. The reduction agent solution is automatically metered by a 1-channel hose pump.
HS 60	Hg/hydride system for flow injection operation with electrically heated cell unit Sample and reduction agent solution, acid and waste are transported through the system by means of hose pumps. The AS-F and AS-FD autosamplers can supply the sample solutions to the Hg/hydride system in a fully automatic manner.

All Hg/hydride systems allow the detection of the hydride-forming elements and mercury using the hydride and cold vapor methods.

You can extend the functionality of the HS 55 and HS 60 Hg/hydride systems with the following accessories and modules:

Module	Function
Hg cell ("tulip")	Tulip-shaped cell for higher sensitivity for Hg detection
Hg Plus Upgrade Modul	Enrichment of mercury on a gold collector for ultra-trace analysis
HydrEA Upgrade Kit	Coupling of the Hg/hydride systems to the graphite tube AAS for ultra-trace analysis of the hydride-forming elements and mercury An AS-GF autosampler must be used to supply the samples to the AAS.

You can retrofit these accessories and modules yourself.

3.2 Design

The Hg/hydride system consists of a basic module, the functional module and an optional module for mercury enrichment. The three modules are plugged on top of each other and electrically connected through mixed plug connectors.

The Hg/hydride system can be used with the following AAS devices:

- ZEEnit 700 P, ZEEnit 700 Q, ZEEnit 650 P
- contrAA 800 series
- novAA 800 series, novAA 350i

For information on using the Hg/hydride systems with older Analytik Jena AAS models, please contact customer service.

Detailed information on the design of the AAS devices and on the analysis process can be found in the AAS device manuals.

Fig. 1 Hg/hydride system with AAS device

All functionally relevant assemblies are located on the front plate of the batch functional module:

- 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.

Fig. 2 Front view with main assemblies

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

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

The optional mercury enrichment module is inserted into the functional module from above and electrically connected to it. The hoses are routed to the frame of the functional module and from there to the front plate. When changing between the "Hydride (batch)"/"Hg without enrichment (batch)" and "Hg with enrichment (batch)" modes, you only change the hose routing on the front plate.

Supply the sample manually into the Hg/hydride system. To do so, pipette a volume of 1 to 20 ml into the reaction beaker and clamp the reaction beaker to the head of the batch module with a slight rotation so that it is gastight.

After the analysis has started, the 1-channel hose pump meters the reduction agent automatically into the reaction beaker. The fast and partly vigorous reaction releases gaseous metal hydride or atomic Hg vapor. Hydrogen is formed as a by-product during the reaction with $NaBH_4$.

Argon is used as the carrier and purge gas. The argon flow transports the reaction products either directly to the AAS device or to a gold collector for Hg enrichment. The enriched mercury is released during the bake-out of the gold collector and transported to the atomizer by a directly connected argon flow.

Remove the reaction beaker after the measuring process, rinse it and refill it with sample.

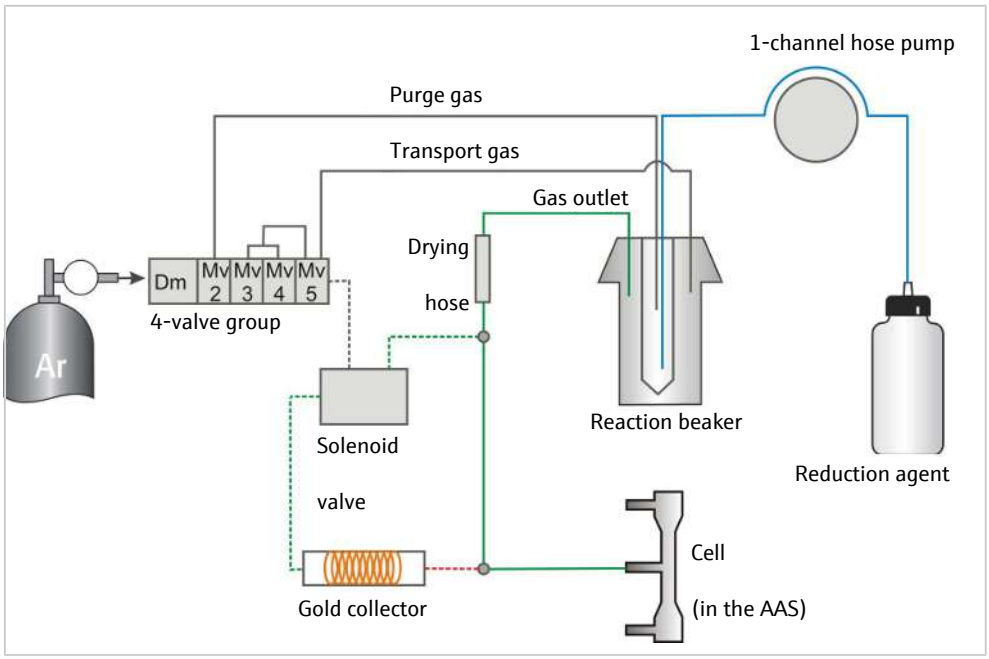


Fig. 3 Functional diagram

The Hg/hydride system normally works with sodium borohydride ($NaBH_4$) as the reduction agent. Alternatively, you can use tin(II) chloride ($SnCl_2$) for Hg detection.

NOTICE

High workload when changing the reduction agent

Changing the reduction agent requires major maintenance work.

- Renew all hoses that have come into contact with the reduction agent.
- Flush the Hg/hydride system thoroughly before loading samples the next time.

3.2.1 Hose pump

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

3.2.2 Batch module

The batch module consists of a reaction beaker with a conical bottom for sample volumes of 1 to 20 ml and the head.

The head contains:

- purge gas supply (marked "purge gas" at the head), argon gas flow 15 l/h
- transport gas supply (marked "transp. gas"), argon gas flows 6 l/h and 25 l/h
- gas outlet to the cell (marked "to cell")
- flange seal for reaction beaker
- Metering tip

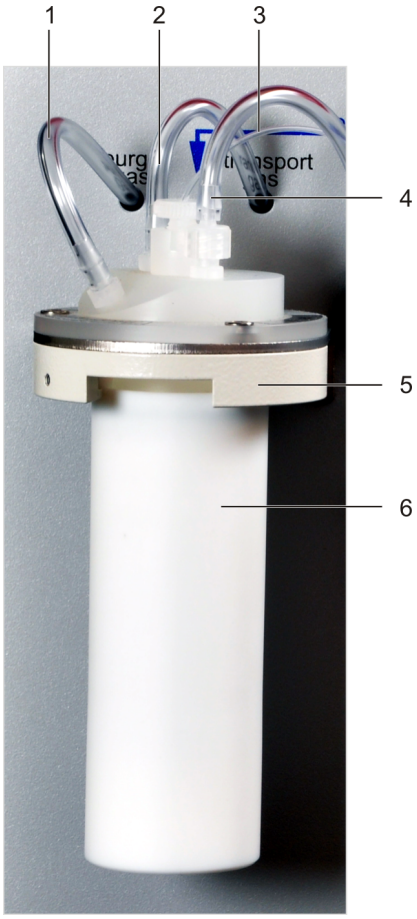


Fig. 4 Batch module

1 "purge gas" gas supply
2 "transp. gas" gas supply
3 Metering hose for reduction agent
4 "to cell" gas outlet
5 Head
6 Reaction beaker

Reduction agent and purge gas are transported through the metering tip to the bottom of the reaction beaker. The reaction with the sample initially takes place in the lower part of the reaction beaker. The released reaction gas and the purge gas accelerate the reaction. The purge gas drives the released metal hydride or Hg vapor out of the sample solution. The transport gas ("transp. gas") enters the reaction beaker at the top. The transport gas transports the metal hydride or the Hg vapor from the reaction beaker to the atomizer in the AAS device or to the gold collector.

3.2.3 4-valve group for gas control

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

Valve MV2	Gas flow F2 with a flow of 15 l/h ■ Constant purge gas flow through the pipette tip of the batch module
Valves MV3/MV4	Gas flow F3 with a flow of 6 l/h and gas flow F4 with a flow of 25 l/h ■ Transport gas flow: the flows can be selected individually or as a total gas flow (31 l/h).
Valve MV5	The valve switches gas flows F3 and F4 either to the batch module or to the gold collector to expel the released mercury.

3.2.4 Hg Plus Upgrade Modul (optional)

The optional module is located in a compartment at the top of the Hg/hydride system. You can open the red cover of the Hg/hydride system and access the module.

The module consists of the following components:
- quartz tube with the gold collector
- infrared sensor
- fan
- 3/2-way solenoid valve (at the inlet)

The solenoid valve switches the following gas flows to the gold collector:
- reaction gas for loading the gold collector
- direct gas flow for expelling the enriched mercury

The gold collector is the core of the module. It contains a loosely rolled-up gold/platinum mesh which is approx. 20 mm wide. The mesh is secured in the quartz tube.

The gold collector enriches the mercury from the reaction gas on its surface by means of amalgamation. Mercury is only released again from the gold collector when heated to a temperature of approx. 630 °C during bake-out.

The quartz tube is surrounded by a heating coil. The heating coil supplies heat to the gold collector from the outside during bake-out. An infrared sensor monitors the temperature. After bake-out, the axial fan cools the quartz tube with air. This way, the Hg/hydride system prepares the gold collector for the next measurement.

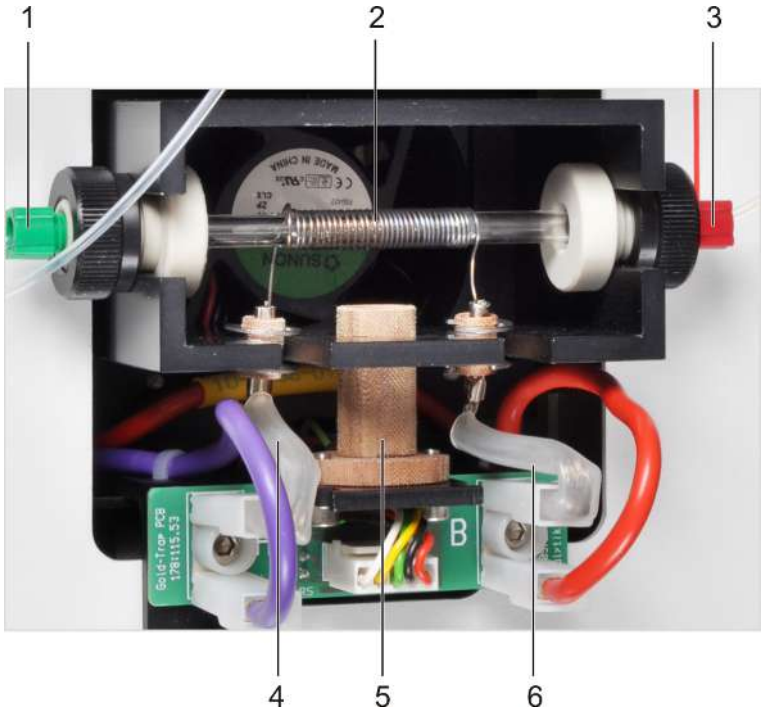


Fig. 5 Gold collector

1 Gas inlet
2 Heating wire
3 Gas outlet
4 Heating connection
5 Infrared sensor
6 Heating connection

3.2.5 Type plate

The type plate is located on the rear of the device. The type plate contains the following information:

- company name and complete address of the manufacturer
- device designation: type designation and trade name
- model and serial number

3.3 Measuring process

You can start the measurement either by pressing the "Start" button on the Hg/hydride system or via the control and analysis software of the AAS.

Start the measurement immediately after you have filled a new sample into the reaction beaker and attached the reaction beaker in a gastight manner. The Hg/hydride system first purges the reaction beaker with argon to remove any air. After a certain waiting time, the software registers the zero value (AZ = Auto Zero).

After the baseline adjustment, the hose pump starts pumping reduction agent into the reaction beaker. After a short time, the purge flow starts: the carrier gas argon transports the metal hydrides formed or the elementary mercury to the AAS. Atomic vapor forms in the quartz cell or the graphite tube furnace, respectively. The metal hydrides decompose due to collisions.

In mercury detection, the pre-wash time and the AZ waiting time are omitted to prevent the highly volatile mercury from being expelled from the sample too early. The zero value is determined over a period of 2 s instead of 4 s.

Measuring process with enrichment

Before a sample is supplied to the atomizer of the AAS device, the Hg/hydride system enriches mercury from one or more samples on the gold collector. The gold collector releases the mercury again during bake-out. With the subsequent cooling, the Hg/hydride system prepares the gold collector for the next measurement.

Operation times

In the control and analysis software, you can define the following operation times in a method for the Hg/hydride system:

Option	Description
Prewash time	Time for purging the beaker with argon before the reaction (for hydride formers) The pre-wash time is used to expel air to prevent an oxyhydrogen reaction during the subsequent reaction.
Pump time	In this time, the reduction agent is pumped into the beaker in order to start a reaction.
AZ wait time	Time directly preceding the baseline adjustment (AZ = auto zero).
Wash time	The wash times are needed to transport the reaction gas with the argon flow. The transport paths can be graphically displayed.
Heat. time collector	During this time, the heater runs to release the enriched mercury from the gold collector.
Cool. time collector	During this time, the gold collector is cooled to make it ready for the next enrichment.

Graphical display

To open the graphical display of the gas paths, click on **Plot** in the **Method** | **Hydride** window. The window displays the operations that run partially simultaneously during the individual phases of the measurement.

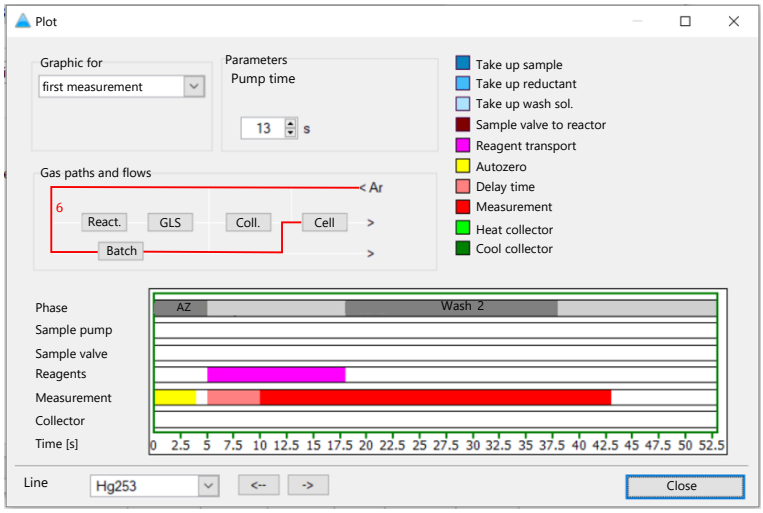


Fig. 6 Graphical display: gas paths and measuring process

4 Installation and commissioning

Normally, the Hg/hydride system is installed by the Analytik Jena customer service or persons authorized by Analytik Jena together with the AAS device. If the Hg/hydride system is delivered separately at a later time, you can also install it yourself.

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

To ensure trouble-free operation, please make sure that the installation conditions are observed. To do so, check the installation conditions specified in the manual for your AAS device.

4.1 Installing the hydride and Hg cold vapor systems

NOTICE

Continuous beeping tone in the case of incomplete installation

If the installation is incomplete, the device emits a continuous beeping tone.

- In this case review the installation steps carried out.

Information on the AAS device can be found in the separate operating manual.

4.1.1 Installing the cell unit

WARNING

Risk of oxyhydrogen formation

The cell must be sealed gastight for the hydride system as otherwise an oxyhydrogen mixture which could explode at high temperatures would form in the cell.

- Inspect the polished end faces of the cell.
- Even if you notice only minor damage, replace the cell.

NOTICE

Risk of corrosion

If acid residues remain in the siphon of the mixing chamber/nebulizer system, there is a risk of corrosion of the cell unit due to the acid vapors.

- Flush the siphon with 500 ml of water via the mixing chamber connection before placing the cell unit on the mixing chamber connection.

- ▶ Remove the burner head from the burner block.
- ▶ Flush the siphon with 500 ml of water via the mixing chamber neck.
- ▶ Attach the cell unit to the burner block and lock it.

- Fold the cell unit upwards. Insert a suitable cell:

Fig. 7 Quartz cell for hydride method

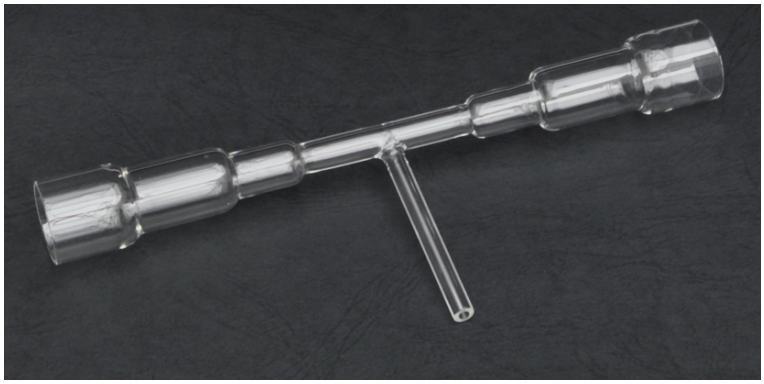


Fig. 8 Hg cell (made of quartz, tulip-shaped)

- For the **hydride method**: Insert the quartz cell.
- Close and lock the cell unit.
- Attach the frames with the quartz windows on both sides and clamp them in place with the leaf springs.
- Attach the gas hose coming from the Hg/hydride system to the middle connection of the cell. The gas hose connection on the Hg/hydride system is marked "to cell".
- Also attach the gas discharge hose to the outer connections. If possible, hook the T-piece into the sample chamber panel at the rear of the sample chamber.
- Route the gas discharge hose into the extractor.
- For the **Hg cold vapor method**: Insert the Hg cell.
- Close and lock the cell unit.
- Secure the Hg cell with the tension springs to lock it in its position.
- Attach the gas hose coming from the Hg/hydride system to the middle connection of the cell. The gas hose connection on the Hg/hydride system is marked "to cell".

  ✓ The cell unit has now been installed in the AAS device.

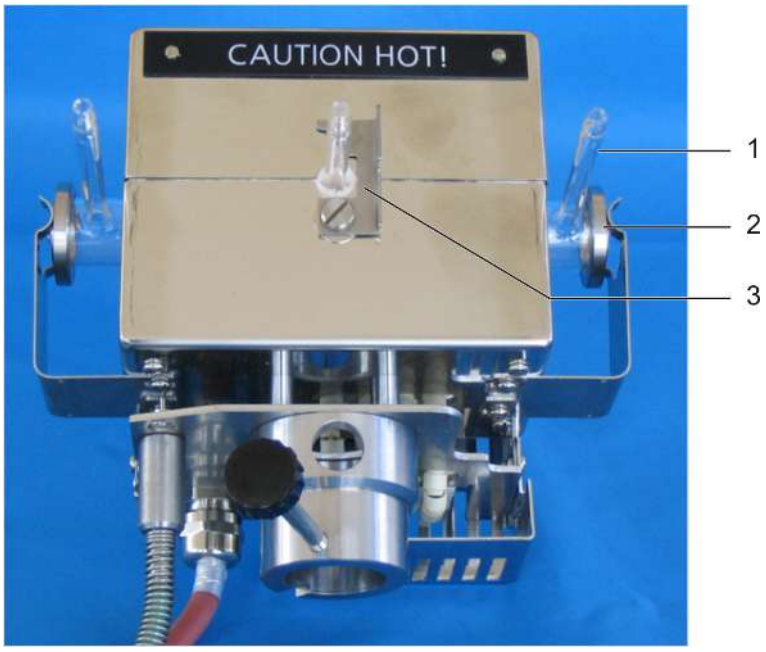


Fig. 9 Cell unit with quartz cell

1 Quartz cell for hydride method
2 Frame with quartz window
3 Lock

Installing the cell unit on the ZEEnit 650 P

The ZEEnit 650 P model is only equipped with a graphite tube furnace. In this model, the graphite tube furnace can be pulled out of the sample chamber and the cell unit for the Hg/hydride method can be installed.

- Unscrew the attachment screw on the front under the graphite tube furnace.
- Pull the graphite tube furnace out of the sample chamber.
- Lock the furnace plate in the pulled-out position with the locking pin.

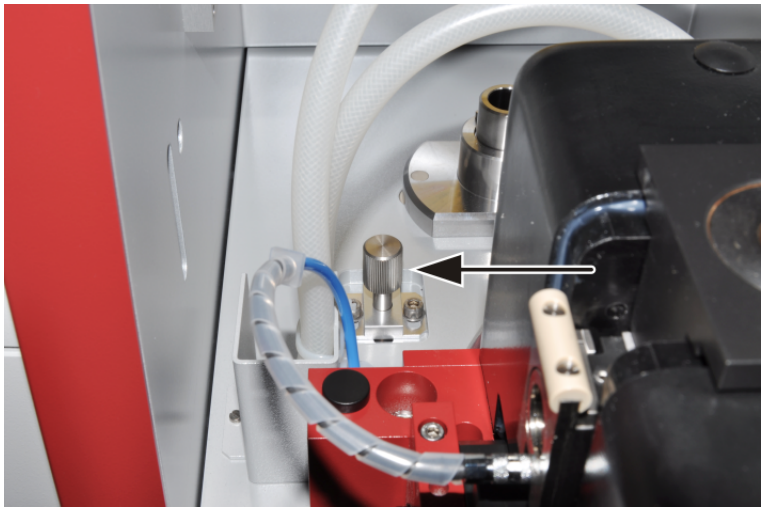


Fig. 10 Locking pin on the furnace plate

- Insert the receptacle for the cell unit into the corresponding sockets on the floor plate of the sample chamber.
- Attach the cell unit to the receptacle and lock it with the attachment screw.

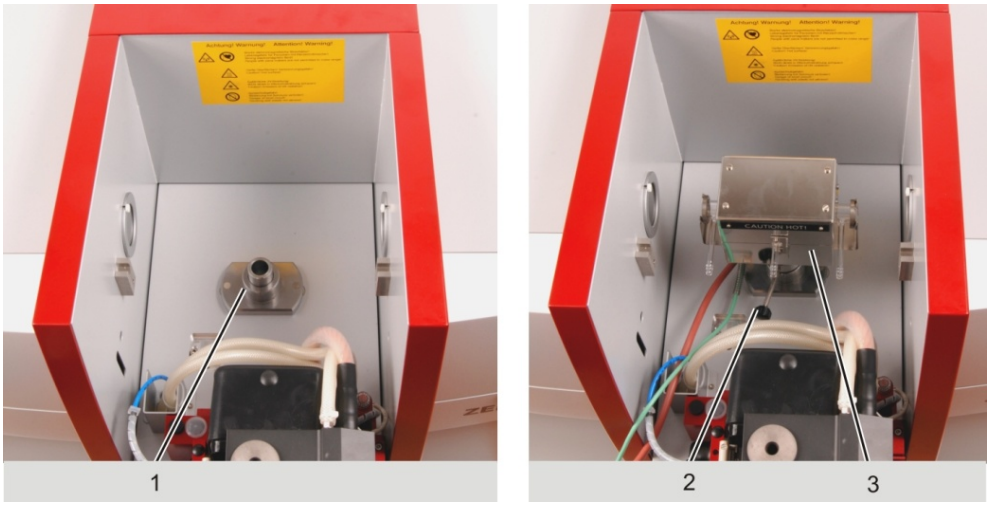


Fig. 11 Receptacle and cell unit for Hg/hydride system

1 Receptacle for cell unit
2 Attachment screw
3 Cell unit

- Insert the corresponding cell into the cell unit and proceed as described at the beginning of this chapter.

4.1.2 Installing the Hg/hydride system on the AAS

CAUTION

Dangerous voltage at the cell heating connection

Dangerous voltage may be present at the cell heating connection.

- Only connect the device and the other components to the power grid when they are switched off.
- Only connect and disconnect electrical connection cables between the system components when the system is switched off. Otherwise the sensitive electronics may also get damaged.

Fig. 12 Hg/hydride system installed on the AAS

- Place the Hg/hydride system to the right of the AAS device or onto a table next to the AAS device.

Electrical connection and interfaces

- Connect the cell unit electrically to the Hg/hydride system:
  - Connect the heating cable to the "cell heating" connection.
  - Connect the temperature sensor cable to the "cell sensor" connection.
  - Fasten the grounding of the sensor cable on the connection panel of the Hg/hydride system with the knurled head screw.
- Connect the functional module to the AAS: the AAS device provides the voltages (+5 V/+24 V) for the functional module.
  - Connect the "AAS" plug of the twin cable to the AS or Sampler Flame socket on the AAS device.
  - Connect the "HS" D-sub socket of the thinner cable of the twin cable to the "input 5 V/24 V" connection of the Hg/hydride system.
  - The round green "AS" socket of the twin cable is not used.
  - Connect the signal cable to the HS or the Hydridsystem connector of the AAS device. Connect the other end of the cable to the "AAS/RS 232" interface of the Hg/hydride system.

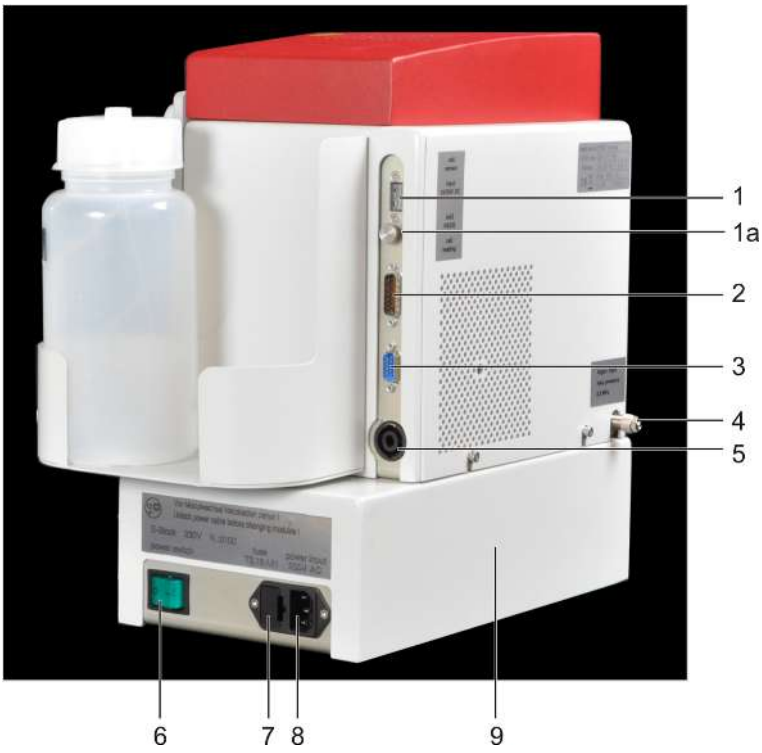


Fig. 13 Connections on the right of the device

1	Cell unit temperature sensor connection ("cell sensor")	1a	Knurled screw for grounding
2	Connection for +5 V/+24 V supply from the AAS	3	"AAS/RS 232" interface
4	Argon connection	5	"cell heating" connection
6	Power switch	7	Fuse holder
8	Power connection	9	Basic module

- Connect the power cable to the power connection on the basic module. Use the multiple socket provided with the AAS device for the connection.
- Connect the Argon hose to the bulkhead fitting on the rear.

"Hydride"/"Hg without enrichment" mode

- Select the drying hose: "Hy" for the hydride method or "Hg" for mercury detection.
- Connect the cell hose to the drying hose via the female Luer coupling.
- Connect the drying hose via the connection hose to the "to cell" gas outlet of the batch module.
- If not already done: attach the cell hose to the middle connection of the cell.

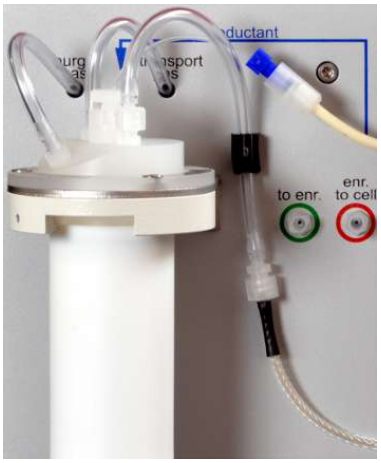


Fig. 14 Hose routing for the "Hydride"/"Hg without enrichment" mode

"Hg with enrichment" mode

- Select the drying hose type "Hg".
- Connect the drying hose to the gas connection marked "to cell" on the batch module via the connection hose.
- Screw the other end of the hose to the connection marked "to enr." (to enrichment) on the front plate.
- Attach the cell hose to the connection marked "enr. to cell" on the front plate.
- If not already done: attach the cell hose to the middle connection of the cell.

Fig. 15 Hose routing for the "Hg with enrichment" mode

Reduction agent transport

- Fill the storage bottle with the reduction agent.
- Connect the reduction agent intake hose (with the blue hollow screw) to the pump hose of the 1-channel pump and immerse it into the storage bottle for the reduction agent up to the stopper.
- Hook the hose cartridge into the Hg/hydride system. Set the locking levers so that the solution is transported evenly.
  - ✓ The Hg/hydride system is now installed on the AAS device and ready for measurements.

Activation sequence

The AAS device supplies the operating voltages of +5 V/+24 V to the functional module. Line voltage is only present at the basic module. During the activation initialization, the functional module checks the line frequency. If no voltage is present at the basic module, the functional module cancels the initialization.

This leads to the following activation sequence:

- Switch on the Hg/hydride system.
- Switch on the AAS device.
  - ✓ First measurements can be started.

4.1.3 Changing between operating modes

To change between the "Hydride" or "Hg without enrichment" and "Hg with enrichment" modes, you have to change the hose routing on the front plate of the batch module.

"Hydride"/"Hg without enrichment" mode

- Select the drying hose: "Hy" for the hydride method or "Hg" for mercury detection.
- Connect the cell hose to the drying hose via the female Luer coupling.
- Connect the drying hose via the connection hose to the "to cell" gas outlet of the batch module.
- If not already done: attach the cell hose to the middle connection of the cell.

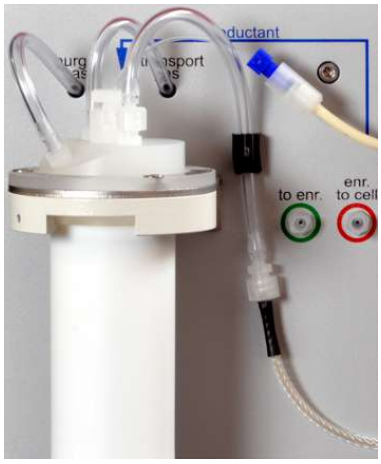


Fig. 16 Hose routing for the "Hydride"/"Hg without enrichment" mode

"Hg with enrichment" mode

- Select the drying hose type "Hg".
- Connect the drying hose to the gas connection marked "to cell" on the batch module via the connection hose.
- Screw the other end of the hose to the connection marked "to enr." (to enrichment) on the front plate.
- Attach the cell hose to the connection marked "enr. to cell" on the front plate.
- If not already done: attach the cell hose to the middle connection of the cell.

Fig. 17 Hose routing for the "Hg with enrichment" mode

Selecting the cell

- **"Hydride"** mode: use the quartz cell.
- Insert the cell into the cell unit of the AAS device and close it with the quartz windows.
- **"Hg with/without enrichment"** modes: optionally select the tulip-shaped Hg cell. Alternatively, the quartz cell can be used.
- Insert the cell into the cell unit and close it with the springs.

4.2 Retrofitting the Hg Plus Upgrade Modul

WARNING

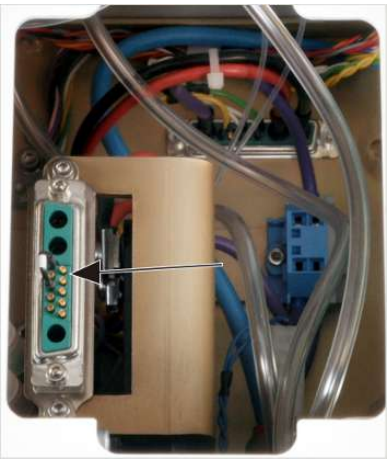
Risk of electric shock

There is a risk of touching live components and suffering an electric shock when converting the Hg/hydride system.

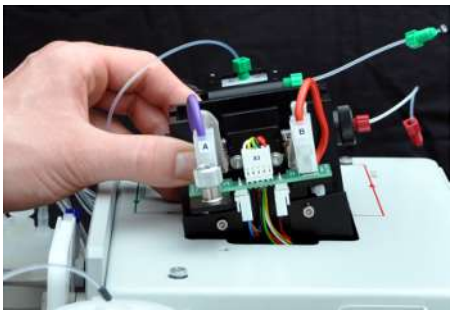
- Before the conversion: switch off the Hg/hydride system and the AAS at the power switch.
- Disconnect the power plug from the socket.
- Disconnect all connection cables to the AAS and the cell unit.

Software tool for the conversion

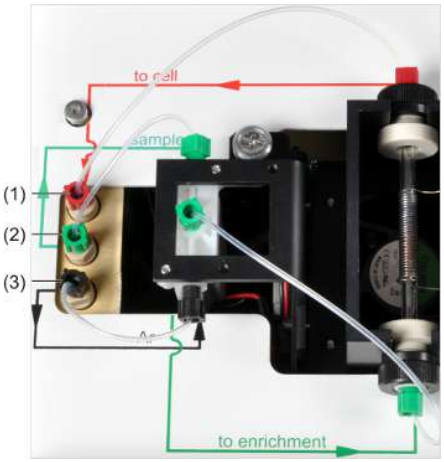
- Insert the CD included in the delivery into the PC. Start the HS Wizard program and follow the on-screen instructions.
- Select the spectrometer used.
- Select the starting configuration of the Hg/hydride system.
- Select the target configuration of the Hg/hydride system with Hg enrichment.



- Open the red hood of the Hg/hydride system.
- Pull the short-circuit plug in the Hg/hydride system up and remove it.



- Insert the module. To do so, align the module with the guide pins and push it down until the plug-in connection has been established.
- Secure the module with the knurled head screws.



- Make the hose connections to the Hg/hydride system via the frame:
  - Attach the hose with the red hollow screws to the rear connection with the red arrow (1).
  - Attach the hose with the green hollow screws to the middle connection with the green arrow (2).
  - Attach the hose with the black hollow screws to the front connection with the black arrow (3).
- Close the red hood.
- Connect the system to the power line, the AAS device and the cell unit.
- Switch on the devices: first the Hg/hydride system, then the AAS.
- After the devices have been initialized in the HS Wizard program, click the **[Next]** button. Exit the program.

Functional test

- Start the ASpect LS or ASpect CS program.
- In the **Quick Start** window, select the **Hydride** method and initialize the device configuration.
- Close the window with **OK**.
- Click the **Hydride system** button.
- Check the heating function. Open the red device hood.
- In the **Hydride system** window, select the **Heating on** option under **Collector** on the **Control** tab.
- Check whether the heating coil lights up.
- Check the cooling. Select the **Cooling on** option.
- Check whether a vertical air flow can be felt.
- End the functional test. Select the **off** option. Close the red hood.

- Close the **Hydride system** window.
  - ✓ The new module is ready for operation.

4.3 Installing the HydrEA system

NOTICE

Error message when the dummy plug is missing

No cell unit is used for the HydrEA method. The Hg/hydride system emits an acoustic signal if the temperature sensor connection is open.

- To ensure proper operation of the device, insert the dummy plug included in the delivery into the temperature sensor connection.

Carry out the installation steps in the following sequence:

- If the Hg/hydride system is already installed: prepare the analysis system for the HydrEA system.
- Install and adjust the graphite tube system and the autosampler for the graphite tube furnace (AS-GF) as described in the user manual for the AAS device.
- Coat the graphite tube with iridium or gold.
- Install the HydrEA system in the Hg/hydride system.
- Adjust the autosampler for the graphite tube furnace with the titanium cannula (for the HydrEA system).

4.3.1 Preparing the analysis system for the HydrEA system

CAUTION

Dangerous voltage at the cell heating connection

Dangerous voltage may be present at the cell heating connection.

- Only connect the device and the other components to the power grid when they are switched off.
- Only connect and disconnect electrical connection cables between the system components when the system is switched off. Otherwise the sensitive electronics may also get damaged.

To convert the analysis system from the hydride system or Hg cold vapor system to the HydrEA system, prepare it as follows:

- Switch off the device and the AAS. Disconnect the power plug from the power outlet.
- Allow the cell unit to cool down.
  ⚠ CAUTION! Risk of burns at the cell unit!
- Disconnect the connection cables for the temperature sensor and the cell heating from the Hg/hydride system.
- Insert the short-circuit plug into the temperature sensor connection.
- If the basic device has only one sample chamber: remove the flame autosampler from the sample chamber and put it next to the Hg/hydride system.
- Disconnect the cell hose from the cell.

- For AAS devices with only one sample chamber: remove the cell and also remove the cell unit.

4.3.2 Coating the graphite tube

NOTICE

Damage to the titanium cannula

Do not coat the graphite tube using the titanium cannula of the autosampler for the graphite tube furnace. Otherwise the cannula can no longer be used for measurements.

- Only coat the graphite tube using the standard configuration of the autosampler, i.e., with the MFA metering hose.

Coat the graphite tube with iridium for detecting hydride-forming elements or with gold for detecting mercury.

Recommendation

Pipette 50 µl iridium or gold master solution (1000 mg/l) into the graphite tube with the autosampler three times in a row and allow the solution to dry on the graphite tube. After the coating furnace program, 150 µg metallic iridium or gold remains on the tube bottom.

Do not exceed a temperature of 2200 °C (iridium) or 1000 °C (gold) during coating and bake-out of the graphite tube. Otherwise iridium or gold loss will occur.

- Start the ASpect LS or ASpect CS program.
- In the **Quick Start** window, select the **Graphite Furnace** (**Wall**) method and initialize the device configuration.
- Close the window with **OK**.
- Click the **Furnace** button.
- Select the **Plot** tab and check the box next to **Graphite tube coating**.
- Define the coating parameters.
  - **Cycles** = number of pipetting cycles (recommended: 3)
  - **Position** = position of the iridium or gold master solution on the plate of the autosampler
  - **Vol.** = sample volume to be pipetted per cycle (recommended: 50 µl)
  - **Element** = Ir or Au
  - ✓ The diagram on the tab shows the fixed temperature/time gradient for tube coating with iridium or gold.
- Place the sample container with the iridium or gold master solution into the selected position on the plate of the autosampler.
- Click **Start** to start the coating process.
  - ✓ The graphite tube is coated with iridium or gold.

Ir coating program

Step	Temperature [°C]	Rate [°C/s]	Hold time [s]	Purge gas
Drying	90	5	40	Max
Drying	110	1	40	Max
Drying	130	1	40	Max
Pyrolysis	1200	300	26	Stop
Atomizing	2100	500	8	Stop
Bake-out	2100	0	5	Medium

Au coating program

Step	Temperature [°C]	Rate [°C/s]	Hold time [s]	Purge gas
Drying	80	5	25	Max
Drying	90	1	25	Max
Drying	110	5	10	Max
Pyrolysis	110	0	6	Stop
Atomizing	950	500	5	Stop
Bake-out	950	0	5	Medium

4.3.3 Installing the Hg/hydride system on the AAS

- At the autosampler for the graphite tube furnace (AS-GF), loosen the clamping nut of the hose guide, pull out the metering hose and deposit it in the waste bottle.
- Insert the titanium cannula for the HydrEA system into the hose guide up to the bend and secure it.
- Place the Hg/hydride system near the AAS.

Electrical connection and interfaces

- On the Hg/hydride system, connect the dummy plug to the cell unit temperature sensor connection.
  NOTICE! Without the dummy plug, the device emits a an acoustic signal.
- Connect the functional module to the AAS: the AAS device provides the voltages (+5 V/+24 V) for the functional module.
  - Connect the "AAS" plug of the twin cable to the AS or Sampler Flame socket on the AAS device.
  - Connect the "HS" D-sub socket of the thinner cable of the twin cable to the "input 5 V/24 V" connection of the Hg/hydride system.
  - The round green "AS" socket of the twin cable is not used.
  - Connect the signal cable to the HS or the Hydridsystem connector of the AAS device. Connect the other end of the cable to the "AAS/RS 232" interface of the Hg/hydride system.

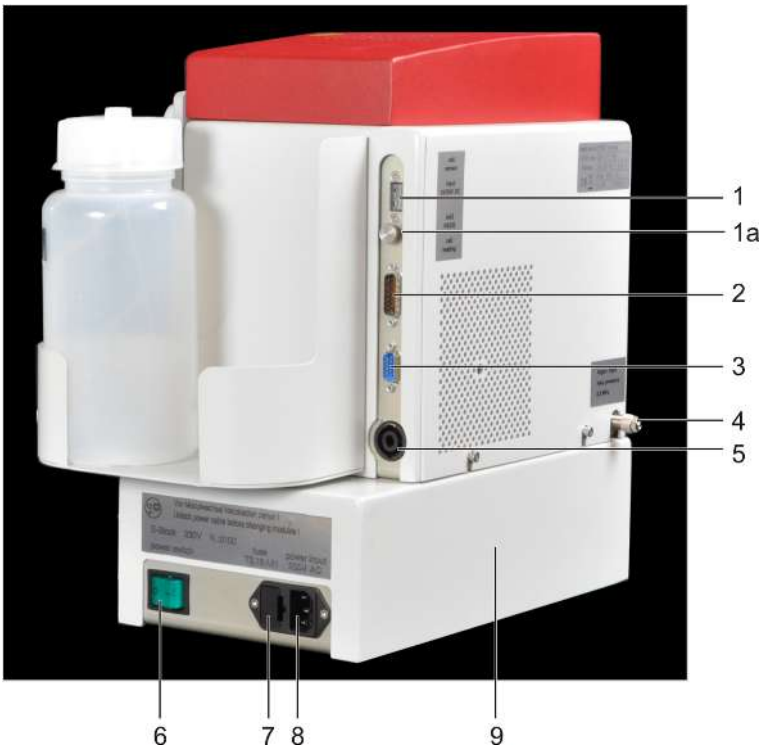


Fig. 18 Connections on the right of the device

1	Cell unit temperature sensor connection ("cell sensor")	1a	Knurled screw for grounding
2	Connection for +5 V/+24 V supply from the AAS	3	"AAS/RS 232" interface
4	Argon connection	5	"cell heating" connection
6	Power switch	7	Fuse holder
8	Power connection	9	Basic module

- Connect the power cable to the power connection on the basic module. Use the multiple socket provided with the AAS device for the connection.
- Connect the Argon hose to the bulkhead fitting on the rear.
- Connect the HydrEA hose to the coupler on the connection marked "to cell" of the batch module and plug it onto the titanium cannula of the AS-GF.

Reduction agent transport

- Fill the storage bottle with the reduction agent.
- Connect the reduction agent intake hose (with the blue hollow screw) to the pump hose of the 1-channel pump and immerse it into the storage bottle for the reduction agent up to the stopper.
- Hook the hose cartridge into the Hg/hydride system. Set the locking levers so that the solution is transported evenly.

  ✓ The Hg/hydride system is now installed on the AAS device and ready for measurements.

Activation sequence

The AAS device supplies the operating voltages of +5 V/+24 V to the functional module. Line voltage is only present at the basic module. During the activation initialization, the functional module checks the line frequency. If no voltage is present at the basic module, the functional module cancels the initialization.

This leads to the following activation sequence:

- Switch on the Hg/hydride system.
- Switch on the AAS device.
  - ✓ First measurements can be started.

4.3.4 Adjusting the autosampler for the graphite tube furnace with the titanium cannula

- Start the ASpect LS or ASpect CS program.
- In the **Quick Start** window, select the **HydrEA** method and initialize the device configuration.
- Close the window with **OK**.
- Click the **Autosampler** button. Select the **Techn. parameters** tab and click the **Align sampler to furnace** button.
  - ✓ The software guides you through the adjustment in the x and y direction and the lowering of the titanium cannula step by step.
- Insert the adjustment aid:
  - novAA 800, contrAA 800: insert the adjustment aid with the crosshair into the pipetting opening.
  - ZEEnit 700 P, ZEEnit 700 Q, ZEEnit 650 P: open the Zeeman furnace and 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.
- Continue following the software instructions:
  - Use the **left** / **right** buttons to align the autosampler in the X-direction (parallel to the optical axis) with the crosshair or the dosing opening. Use the adjusting screws on the sides for fine adjustment.
  - Turn the adjusting screw to perform the adjustment in the Y-direction (sample chamber depth).
  - Tighten the screws and secure the setting with lock nuts.
  - Use the software to set the Z-direction: Lower the titanium cannula until the cannula is flush with the top edge of the adjustment aid.
- Click the **Next** button to save the settings in the software.
- Remove the adjustment aid.
- Prepare the graphite tube:
  - novAA 800, contrAA 800: insert the dosing funnel into the pipetting opening.
  - ZEEnit 700 P, ZEEnit 700 Q, ZEEnit 650 P: insert the left furnace window, insert the standard graphite tube or coated graphite tube, close the Zeeman furnace.
- Adjust the sample injection depth into the graphite tube:
  - Loosen the clamping nut, place the titanium cannula on the tube bottom and check the position of the cannula with the furnace camera if necessary.
  - Secure the cannula with the clamping nut.
  - Set the injection depth above the tube bottom (approx. 0.5 mm).
- Click **Finish** to complete the adjustment.
  - ✓ The autosampler has now been adjusted and is ready for measurements.

5 Operation

How to prepare the AAS device for the hydride or HydrEA method is described in the user manual for the AAS device.

The software manual describes the following:
- how to set the method and the operating mode
- how to check the functionality of the Hg/hydride system
- how to set a hydride or HydrEA method
- how to set the method parameters for the autosampler

5.1 Preparing operating materials and standards

WARNING

Risk of chemical burns

Sodium borohydride and sodium hydroxide are highly corrosive, hygroscopic and, in solution, extremely aggressive. Concentrated hydrochloric acid is highly corrosive.

- Wear protective goggles and protective clothing when handling these corrosive substances. Work under an extractor.
- Observe all instructions and specifications in the safety data sheets.
- When preparing diluted acids and lyes, always fill in water first. Only then add concentrated acid or lye.

WARNING

Risk of poisoning due to arsenic

The arsenic standard solution (1000 mg/l) causes severe skin and eye irritation. The solution is carcinogenic.

- Wear protective goggles and protective clothing when handling this hazardous substance.
- Observe all instructions and specifications in the safety data sheets.
- When preparing diluted acids, always fill in water first and slowly stir in the concentrated standard solution.

The following solutions are required for the operation of the Hg/hydride system:
- reduction agent ($NaBH_4$/NaOH)
- standard solutions for the hydride method
- arsenic standard solutions for the HydrEA method
- reduction solution (e.g., KI/ascorbic acid) for reducing As(+V) to As(+III)

Samples can contain arsenic ions in various oxidation states. Treating the sample is therefore recommended to achieve a better measuring sensitivity and a correct measurement.

Recommendation:
- Treat the samples with the reduction solution (KI/ascorbic acid) before transferring them to the Hg/hydride system to ensure that arsenic is available in the oxidation state +III. Make sure that As(+V) is completely converted to As(+III).

- If possible, treat standards and samples identically. When arsenic standards age, As(+V) ions form in the solution due to reaction with atmospheric oxygen.

Suggestion for preparing the solutions

Reduction agent

Solution	Preparation	Shelf life, comments
Master solution 3.0 % $NaBH_4$ + 1.0 % NaOH	Dissolve 7.5 g $NaBH_4$ + 2.5 g NaOH (pellets) + 250 ml H_2O (dist.) in an ultrasonic bath	Approx. 4 ...6 weeks (refrigerated at ≤7 °C)
Reduction agent 1.0 % $NaBH_4$ + 0.3 % NaOH	100 ml of the master solution + 200 ml H_2O (dist.)	Approx. 1 ...2 weeks (refrigerated at ≤7 °C)

Hydride method standards

Solution	Preparation	Shelf life, comments
Solution 1 (commercial standard solution) 1000 mg/l As		See manufacturer specifications
Solution 2 1 mg/l As	100 µl of solution 1 + 7 ml HCl (37 %, p. a.) Fill up with H_2O (dist.) to 100 ml.	Stable for several days. Prepare standards by means of a dilution series.
Standards 0 / 2.0 / 4.0 / 6.0 / 8.0 / 10.0 µg/l As	Example: 10 µg/l As standard 1 ml of solution 2 + 7 ml HCl (37 %, p. a.) + 1 ml KI/ascorbic acid solution After 45 minutes, fill up with H_2O (dist.) to 100 ml.	Prepare the standards fresh on a daily basis.

HydrEA method standards

Solution	Preparation	Shelf life, comments
Solution 1 (commercial standard solution) 1000 mg/l As		See manufacturer specifications
Solution 2 10 mg/l As	1 ml of solution 1 + 7 ml HCl (37 %, p. a.) Fill up with H_2O (dist.) to 100 ml.	Prepare standards by means of a dilution series.
Solution 3 100 µg/l As	1 ml of solution 2 + 7 ml HCl (37 %, p. a.) Fill up with H_2O (dist.) to 100 ml.	4 ... 5 days

Solution	Preparation	Shelf life, comments
Standards 0 / 0.2 / 0.4 / 0.6 / 0.8 / 1.0 µg/l As	Example: 1 µg/l As standard 1 ml of solution 3 + 7 ml HCl (37 %, p. a.) + 1 ml KI/ascorbic acid solution After 45 minutes, fill up with H_2O (dist.) to 100 ml.	Prepare the standards fresh on a daily basis.

KI/ascorbic acid solution

Solution	Preparation	Shelf life, comments
5 % KI + 5 % ascorbic acid	2.5 g KI + 2.5 g ascorbic acid Fill up with H_2O (dist.) to 50 ml.	Several days (refrigerated at ≤7 °C) Do not use if you notice any signs of discoloration.

5.2 Switching the device on and off

Tasks for daily commissioning

- Hook the hose cartridge for the sample into the 1-channel hose pump.
- Firmly connect the pump hose/pump hoses by adjusting the locking lever.
- Load the system with reduction agent:
  - Start the control and analysis software.
  - In the **Quick Start** window, select the **Hydride** method and initialize the device configuration. Close the window with **OK**.
  - Click the **Hydride syst.** button.
  - Select the **Control** tab and click the **Load system** button.
  - ✓ The device is ready for operation.

Tasks prior to daily shutdown

Before closing, the program asks you whether you would like to clean the Hg/hydride system.

- Click **Start** to start the system cleaning process.
- When prompted, dip the intake hose(s) into distilled water or, alternatively, a slightly acidic solution.
- The Hg/hydride system flushes the reduction agent hose.
- When prompted, remove the hose from the flushing solution. The Hg/hydride system then pumps out the hose.
- If necessary, repeat the system cleaning process.
- Relieve the pump hose by releasing the hose cartridge at the 1-channel pump.
- Recommendation: Store the reduction agent solution in the refrigerator.
  - ✓ The device can be switched off.

6 Maintenance and care

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

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

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

6.1 Maintenance overview

Basic device

Maintenance interval	Maintenance task
Weekly	Visually inspect the pump hoses and the hose path for wear, dirt and deformation. If necessary, replace the hoses.
As necessary and when the analytical performance drops	■ Clean the cell and the cell windows. ■ Replace the hose dryer. ■ Replace the gold collector (Hg Plus Upgrade Modul). ■ Clean the coated graphite tube or evaporate the metal coat (HydrEA Upgrade Kit) and re-coat the tube. ■ Replace the device fuses.
When changing the reduction agent	■ Renew all hoses that have come into contact with the reduction agent. ■ Flush the system thoroughly.

6.2 Replacing the fuses

WARNING

Risk of electric shock

High voltages are present in the interior of the device, which can lead to electric shock if contacted.

- Before opening: Switch off the device via the power switch.
- Disconnect the power cable from the socket.

The power input fuses are located on the right-hand side of the basic module and are labeled. You can replace the fuses yourself.

Fuses

Fuse number	Fuse type for 220 to 230 V line voltage	Fuse type for 100 to 110 V line voltage
F1	T 3.15 A H	T 6.3 A H
F2	T 3.15 A H	T 6.3 A H

6.3 Cleaning the cell and the cell windows

Clean the cell and the cell windows when the measured lamp energy, i.e., the number of counts, drops. In devices with a Zeeman furnace, contamination can be detected by an increase in the PMT voltage.

WARNING

Risk of chemical burns due to hydrofluoric acid

Hydrofluoric acid is highly corrosive and toxic. Danger to life due to swallowing, skin contact or inhalation.

- The operator must wear a rubber apron, gloves and a face mask when handling hydrofluoric acid. Work under an extractor.
- Observe all instructions and specifications in the safety data sheet.

WARNING

Risk of oxyhydrogen formation

The cell must be sealed gastight for the hydride system as otherwise an oxyhydrogen mixture which could explode at high temperatures would form in the cell.

- Inspect the polished end faces of the cell.
- Even if you notice only minor damage, replace the cell.

CAUTION

Risk of burns

The cell is very hot directly after operation: 600 to 950 °C (metal hydrides) and 150 °C (mercury).

- Allow the cell unit to cool down before maintenance.
- Check the current cell temperature in the **Hydride syst.** window on the **Control** tab.

NOTICE

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

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

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

Cleaning the cell windows

- ▸ After the cell unit has cooled down: press the blade spring together and remove the cell windows with the frames.
- ▸ Clean the cell windows with diluted hydrochloric acid.

- Then flush the cell windows with distilled water and allow them to dry without any residues, e.g., by purging them with an inert gas.

  ✓ The cell windows have been cleaned.

Cleaning the cell

- After the cell unit has cooled down: unlock and unfold the cell unit.
- Remove the cell and pull off the hoses.
- Clean the cell for 5 to 10 min in cold, concentrated hydrofluoric acid HF (40 %).
  ⚠ WARNING! Hydrofluoric acid is highly corrosive and toxic.
- Remove the detached dirt film from the inside of the tube by intensive brushing with a round brush under running water.
- Flush the cell with distilled water and allow it to dry without any residues, e.g., by purging.
- Place the cell into the cell unit. Lock the cell unit.
- Attach the cell windows with frames on both sides of the cell and clamp them in place with the blade springs. Check whether the cell windows seal the cell tightly.

  ✓ The cell has been cleaned.

6.4 Inspecting and replacing the pump hose

CAUTION

Risk of chemical burns due to basic solution

The hoses contain the basic reduction agent solution.

- Prior to any maintenance work, close the software and clean the system as suggested. The Hg/hydride system flushes the hoses and pumps them out after removal from the flushing solution.

Regularly inspect the pump and metering hoses for the reduction agent visually for wear, stubborn contamination and deformation. If excessive wear or contamination is detected, the complete hose set for the reduction agent (intake hose, pump hose, metering hose) must be replaced.

Note that the hoses also have to be replaced when the reduction agent is changed ($NaBH_4$ - $SnCl_2$).

- Release the clamping screw at the batch module and pull out the metering hose (MFA).
- Unhook the hose cartridge, remove the pump hose.
- Insert the new pump hose into the cartridge. Note the pumping direction.
- Hook in the hose cartridge and press it on.
- Feed the new metering hose (MFA) into the batch module. Tighten the clamping screw firmly.
  ℹ NOTICE! The hose end must be approx. 10 mm above the pipette tip in the batch module.
- Insert the intake hose into the storage bottle for reduction agent up to the stopper.

  ✓ The new hose set is ready for operation.

6.5 Inspecting and replacing the flange seal in the batch module

After an extended operating time, the sealing ring at the head of the batch module may lose its elasticity and sealing effect and must be replaced.

- Turn the reaction beaker and pull it off the flange.
- Visually inspect the sealing ring on the flange. If then sealing ring is worn, remove it and replace it with a new sealing ring.
- Slide the reaction beaker onto the flange and turn it to lock it in position.

  ✓ The flange seal has been replaced.

6.6 Replacing the hose dryer

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

- Detach the drying hose from the hose section at the outlet marked "to cell" of the batch module and from the cell hose or the inlet marked "to enr." on the front plate.
- Connect the new drying hose to the hose section at the outlet marked "to cell" of the batch module and the cell hose.
- In the "Hg with enrichment" operating mode, screw the second hose end with the hollow screw to the connection marked "to enr." on the front plate.

  ✓ The new drying hose is ready for operation.

6.7 Replacing the gold collector

CAUTION

Risk of burns

The gold collector is very hot directly after operation (up to 630 °C).

- Allow the gold collector with heating coil to cool before maintenance.

Replace the gold collector

- if the sensitivity is significantly decreased during Hg detection with enrichment.
- if the signals are highly scattered and the reproducibility decreases.
- if the heating value increases.
- if the gold collector does not bake out the enriched mercury completely. If this happens, the measuring intensity is only achieved after several measurements in the case of large concentration differences.

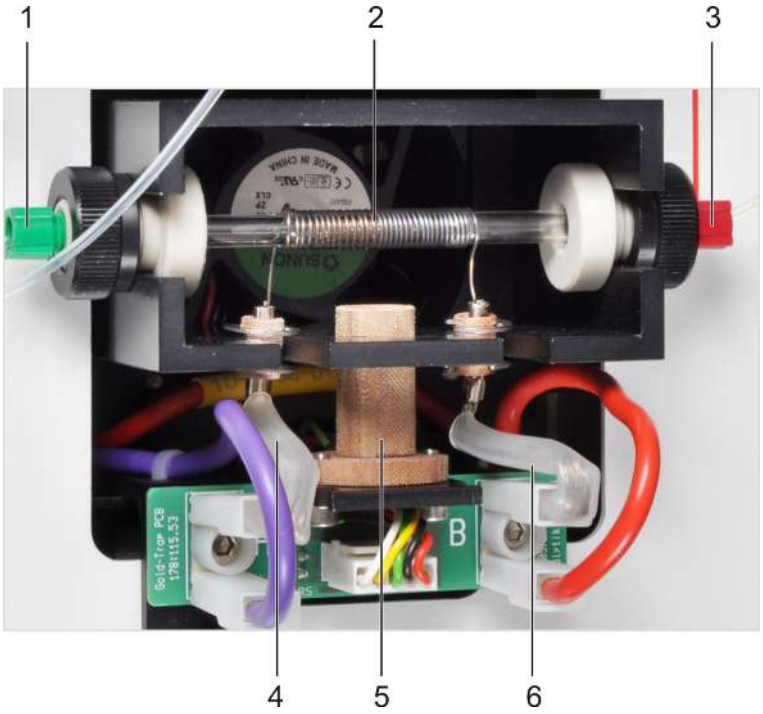


Fig. 19 Gold collector

1 Gas inlet
2 Heating wire
3 Gas outlet
4 Heating connection
5 Infrared sensor
6 Heating connection

- Unscrew the gas inlet and gas outlet hoses from the gold collector.
- Pull the heating coil connections off the PCB.
- Loosen the gold collector screw fitting at the compartment. Remove the gold collector with heating coil and pull off the screw fitting.
- Insert the new gold collector into the screw fitting.
- Insert the gold collector into the compartment. Simultaneously insert the isolation sleeves on the heating wire into the groove.
- Slide the gold collector up to the stop and screw it tight.
- Plug the connections of the new heating coil onto the PCB.
- Tighten the gas hoses on the right and left side of the gold collector with hollow screws.

  ✓ The new gold collector is ready for operation.

6.8 Maintaining the HydrEA system

6.8.1 Cleaning the coated graphite tube

The iridium or gold-coated graphite tube in the HydrEA system can be cleaned by baking out. This procedure can also be repeated multiple times.

The iridium coat evaporates at temperatures above 2200 °C, the gold coat at more than 1000 °C. Do not exceed these temperatures when cleaning.

- Start the ASpect LS or ASpect CS program.

- In the **Quick Start** window, select the **HydrEA** method and initialize the device configuration.
- Close the window with **OK**.
- Click the **Furnace** button.
- Select the **Control** tab and enter the parameters for cleaning the graphite tube in the **Clean furnace** area:
  - **Temp.** = 2200 °C (for Ir) or 1000 °C (for Au)
  - **Ramp** = 500 °C/s (= temperature increase)
  - **Hold** = 10 s
- Click the **Start** button to start the graphite tube bake-out.
  - ✓ The graphite tube is cleaned by a brief bake-out.

6.8.2 Evaporating the iridium or gold coat

A used-up iridium or gold coat can be evaporated by baking out at temperatures ≥2500 °C (Ir) or ≥1800 °C (Au). Attention! The graphite material of the furnace starts to decompose at temperatures of 2600 °C or higher.

You can then re-coat the graphite tube from which the metal coat has been removed for the HydrEA system. You can also use the graphite tube as a standard graphite tube for solution analysis (standard operation).

- Start the ASpect LS or ASpect CS program.
- In the **Quick Start** window, select the **HydrEA** method and initialize the device configuration.
- Close the window with **OK**.
- Click the **Furnace** button.
- Select the **Control** tab and enter the parameters for cleaning the graphite tube in the **Clean furnace** area:
  - **Temp.** = ≥2500 °C (for Ir) or ≥1800 °C (for Au)
  - **Ramp** = 500 °C/s (= temperature increase)
  - **Hold** = 10 s
- Click the **Start** button to start the evaporation of the metal coat.
  - ✓ The metal coat is removed from the graphite tube by baking out.

7 Troubleshooting

Strong foaming can occur in the sample during the hydride and Hg cold vapor methods.

- Test the foaming of unknown samples.
- Immediately stop the measuring process if the transport gas argon transports foam up to the quartz cell.
- Add a few drops of anti-foaming agent to strongly foaming samples, e.g., Dow-Corning DB 110A, silicone anti-foaming agent or octanol.

8 Transport and storage

8.1 Transport

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

Avoid the following during transport:

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

8.1.1 Preparing the device for transport

CAUTION

Dangerous voltage at the cell heating connection

Dangerous voltage may be present at the cell heating connection.

- Only disconnect electrical connection cables between the system components when the system is switched off. Otherwise the sensitive electronics may also get damaged.

CAUTION

Risk of chemical burns

The reduction agent solution contains sodium borohydride and sodium hydroxide and is corrosive.

- Wear protective goggles and protective clothing when handling the corrosive solution.
- Observe all instructions and specifications in the safety data sheets.
- Neutralize the basic solution and dispose of it professionally.
- Also be careful when handling hoses. They can contain residues of the corrosive solution.

- ▶ Exit the control and analysis software and clean the system.
  - ✓ The Hg/hydride system flushes the hoses with distilled water and pumps them out after removal from the flushing solution.
- ▶ Relieve the pump hose by releasing the hose cartridge.
- ▶ Switch off the Hg/hydride system and the AAS. Disconnect the power cable from the socket.
- ▶ Disconnect all connection cables to the AAS and the cell unit.
- ▶ Allow the cell unit to cool down.
  ⚠ CAUTION! Risk of burns at the cell unit!
- ▶ Disconnect the cell hose that connects the Hg/hydride system to the cell unit in the AAS.

- Remove the cell from the cell unit.
- Remove the cell unit.
- Empty and rinse out the storage bottle.
- Turn the reaction beaker to remove it from the batch module.
- Pack open hose ends in protective bags and attach them to the device, e.g., with adhesive tape.
- Carefully package the accessories. Ensure that the glass parts are packed breakproof.
- Package the device and accessories in their original packaging.
  ✓ The device is securely packed for transport.

8.1.2 Moving the device in the laboratory

CAUTION

Risk of injury during transport

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

- Proceed carefully when moving and transporting the device.

Observe the following when moving the device within the laboratory:

- Insufficiently secured components pose a risk of injury!
  Before moving the device, remove all loose parts and disconnect all connections from the device.
- As the device does not have carrying handles, grip the device firmly with both hands at the lower end.
- Observe the guide values and adhere to the legally mandated limits for lifting and carrying loads without auxiliary means.
- Observe the installation conditions at the new location.

8.1.3 Returning the device for servicing

- Clean all device components from biologically hazardous, chemical, and radioactive contamination.
- When registering the return, you will receive a decontamination report from the customer service. Complete the form and attach the signed decontamination declaration to the outside of the shipment.
- Only use the original packaging for the shipment and insert the transport lock. If the original packaging is no longer available, please contact Analytik Jena or your local distributor.
- Attach the warning label to the packaging:
  "CAUTION! SENSITIVE ELECTRONIC DEVICE!".
- Enclose a sheet with the following data:
  - Name and address of the sender
  - Name and telephone number of a contact for inquiries
  - A detailed description of the fault, the precise conditions and situations under which the fault occurs

8.2 Storage

NOTICE

Risk of device damage due to environmental conditions

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

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

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

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

9 Disposal

Auxiliary and operating materials as well as their containers may not be disposed of as domestic waste or enter the sewage system or the soil. The residual liquid from the Hg/hydride system and the autosampler 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 observed.

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

10 Specifications

10.1 Technical data

General characteristics	Designation/type	HS 55
	Dimensions (W x H x D)	360 x 370 x 240 mm
	Mass	14 kg
Procedural data	Mode	Batch mode (discontinuous)
	Detectable elements	As; Bi; Hg; Sb; Se; Sn; Te
	Methods (depending on the configuration)	■ Hydride method ■ Hg cold vapor method without enrichment ■ Hg cold vapor method with enrichment ■ HydrEA method
	Reduction agent	$NaBH_4$ 1.0 % with NaOH 0.3 % Alternatively: $SnCl_2$ 2 to 5 % (only for Hg detection)
Main functional groups	Cell unit	Heating: electric, temperature consistency: ±10 °C Temperature for hydride-forming elements: 600 to 950 °C Temperature for Hg: room temperature or 150 °C
	Absorption cells	Quartz cells with removable quartz windows: length 140 mm, ID 15 mm Hg cell (optional): length 200 mm
	1-channel hose pump	Reduction agent transport Ismaprene hose ID = 2.06 mm, pump speed: adjustable in 4 stages
	Reaction unit	Batch module: PTFE beaker with conical bottom
	Hg Plus Upgrade Modul (optional)	Gold collector: with 0.5 g gold/platinum alloy AuPt as a fine mesh Bake-out temperature: 630 °C, controlled Cooling: axial fan
Sample supply	Sample supply to the AAS (HydrEA system)	Autosampler for the graphite tube method AS-GF
Gas supply	Gas, purity	Argon 5.0
	Inlet pressure	600 kPa
	Working pressure	150 kPa

Gas flow	■ Purge gas: 15 l/h ■ Transport gas: 6 l/h, 25 l/h, 31 l/h

Electrical variables

Voltage	Depending on the basic module: 220 to 230 V or 100 to 110 V
Frequency	50/60 Hz
Fuses	G-fuse sets (5 x 20 mm) F1/F2 ■ T 3.15 A H for 220 to 230 V ■ T 6.3 A H for 100 to 110 V
Power consumption (during heating)	650 VA
Power consumption (during continuous operation)	400 VA
Interfaces to the AAS	"input 5 V/24 V" power supply "AAS/RS 232" interface

Ambient conditions

Temperature during operation	+10 to +35 °C
Humidity	≤90 % at 30 °C
Temperature during storage	-40 to +50 °C
Recommended maximum operating altitude	2000 m (above sea level)

10.2 Standards and directives

Protection class and protection type

The device is protection class I. The housing is protection type IP 20.

Device safety

The device complies with the following safety standards
- EN 61010-1
- The device has contamination degree 2 and overvoltage category II.

EMC compatibility

The device has been tested for radio interference elimination and interference immunity and fulfills the requirements of EN 61326-1.

EU directives

The device meets the requirements of the directive 2011/65/EU.

The device is designed and tested in accordance with standards meeting the requirements of EU directives 2014/35/EU and 2014/30/EU. The device leaves the factory in a sound condition with regard to technical safety. To maintain this condition and to ensure safe operation, the user must strictly observe the safety and operating instructions contained in this operating manual. For accessories delivered with the device and system components from other manufacturers, the information provided in their respective operating manuals has priority.

Guidelines for China

The device contains substances subject to regulation (according to the directive GB/T 26572-2011). Analytik Jena guarantees that, if the device is used as intended, these substances will not leak within the next 25 years and therefore will not pose a threat to the environment or health within this time period.

11 Revision overview

Version	Effective date	Changes
A	2019-09	First version Note: New version labeling after introduction of the Document Management System (A, B, etc.)
B	2021-01	Change of the company's legal form
C	2021-11	■ Installation on current AAS device models ■ Inclusion of the document in the content management system

Table of figures