

AULA-254 Gold

Automatic Mercury Analyzer for the Laboratory



- Fully automated mercury analysis system
- High performance continuous flow analysis (CFA) technique
- Cold Vapor Atomic Absorption Spectrometry (CVAAS)
- Widest dynamic linear range
- Robust construction
- AULAWIN software with extensive and beneficial functions
- Automatic protective function
- Gold trap integrated
- Extension: Automated Sample Digestion module (ASD) for aqueous samples



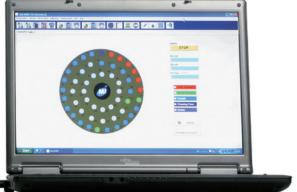


Figure: AULA-254 Gold









Applications

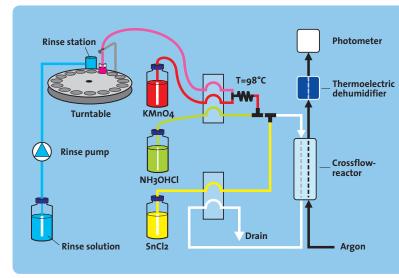
The AULA-254 Gold is a fully automatic analysis system for determining trace mercury levels in water samples and sample digestates. The AULA system is designed to be the right tool for researchers and scientists. The instrument automates routine analysis: it tests sample after sample following a specified procedure exactly. This increases efficiency and productivity in your lab.

Typical applications include:

- Environmental chemistry (water and waste water, effluents, waste, sludge, soil)
- Biological samples (food, urine, blood, saliva, hair)
- Chemical industry (process monitoring, quality control)
- Geochemistry (geological and mineralogical samples)
- Petrochemical samples (oil products)
- Metallurgy and material testing

Reliable and proven method: continuous flow analysis (CFA)

The system's working principle is the continuous flow analysis technique. Sample is continuously drawn from the sample vial and mixed with stannous chloride. This chemically reduces the mercury into the free atomic state. The solution then flows into a special crossflow reactor where an argon stream strips the elemental mercury and carries it into an optical cell, which is placed in the light path of a detector. The cell is entirely made of fused silica and is heated slightly to avoid water condensation.



Flow diagram of AULA-254 Gold with Automatic Sample Digestion

Optimized mercury detection technique

The AULA-254 Gold uses an atomic absorption detector. The detection technique applied is measuring the resonance absorption at ambient temperature using a wavelength of 253.65 nm.

This analytical method is commonly known as cold vapor atomic absorption spectrometry (CVAAS). Unlike typical multi-element AA systems, the AULA-254 is specially designed to detect and quantify mercury levels. This allows top performance in analytical applications.

The AULA-254 Gold uses an electro-optical stabilized electrodeless mercury discharge lamp (EDL) in connection with solid state UV detectors, resulting in excellent baseline stability and detection limits far lower than those of other AA spectrometers.

GoldTrap preconcentration

The integrated GoldTrap mercury preconcentration module enhances detection sensitivity drastically. Using ultra pure reagents and following the guidelines for mercury analysis at ultra trace levels, the AULA-254 Gold can achieve a measuring range from 1 ppt to 5000 ppt.

Small thermal inertia is an outstanding property of our latest GoldTrap design, achieved through the use of a wafer-thin ceramic substrate. Heating and cooling rates are very fast. Analysis duration is comparable to the direct method.

The GoldTrap is installed inside the photometer, thus not increasing footprint space. The user can select by software if analyses will include a GoldTrap preconcentration step or bypass it. This makes the instrument versatile for any application.

High productivity

The typical duration of a full measurement cycle is 60 ... 180 seconds, depending on the set parameters. Measuring calibration standards, samples and QC standards is fully automatic. No long purging or rinsing procedure is needed, even when samples with



The AULA system is simple to use. Samples

are decanted into glass vials and positioned on the autosampler carousel. No weighing of the samples required! Reagent solution (tin-(II)-chloride) and rinse solution (water) are filled into the corresponding bottles. A keystroke starts automatic measurement. The operator can suspend the automatic cycle at any time to select any sample for measurement or remeasurement. New samples can be added, even during a sample run.

Automatic baseline correction

The stability of the baseline is checked prior to each measurement and the zero point is adjusted automatically. Typical zero drift during a measurement is below 0.0001 absorbance units.



Minimized memory effects

Mercury tends to cling to surfaces, causing a carry-over (memory effect) that can compromise results. AULA systems minimize memory effect by using selected materials for components touched by samples and by heating the optical bench. In addition, the autosampler probe and the sample tubing are rinsed after each sample run. Even samples with concentrations in the upper measuring range do not cause carry-over.

Thermoelectric dehumidifier

Traditional mercury analyzers use desiccant-filled dryer tubes or permeation tubes for removing water vapor from the mercury-loaded carrier gas, and these tubes add a maintenance burden. The AULA system uses a maintenance-free thermoelectric dehumidifier. The gas is cooled below the dew point; excess water condenses on the wall of a small glass tube and flows back into the reactor. Absolutely no liquid carry-over! In contrast to other dryers this device has an extremely small surface, which prevents mercury adsorption.

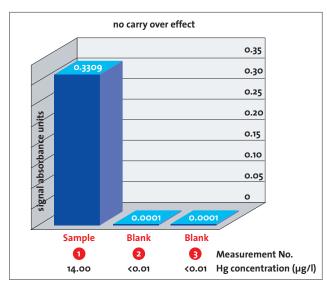


Diagram: blanks measured after a sample with high mercury concentration demonstrating that there is virtually no carry over

Carrier gas flow stabilisation with electronic mass flow controller (MFC)

The stability of the carrier gas flow directly affects the reproducibility of measurements. For this reason the AULA-254 system uses a highly precise (1% accuracy) electronic mass flow controller (MFC) which is built into the photometer. The system saves gas by automatically shutting off flow at the end of the job.

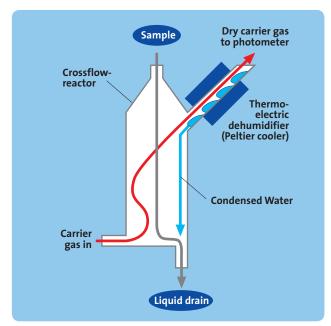


Figure: Schematic of crossflow reactor with thermoelectric dehumidifier

User safety

Release of mercury vapor during mercury analysis is nearly unavoidable. The AULA system collects mercury vapor in a sulfurized activated carbon filter, preventing the vapor from escaping into the working environment. A message appears on the screen if the filter needs replacement. A fume hood is not required.

Automatic protective system cleaning

The instrument automatically interrupts measurement if a sample with a mercury concentration exceeding the safe range is detected. In this case the system immediately performs a cleaning step. The remaining sample can be diluted and used for a second run.

Full-feature software

The AULAWIN software offers the complete feature set of modern analytical software. Developed with substantial input from our users, it is technically mature and easy to use. Samples, calibration standards and quality control standards (QCs) are selected by simple point-and-click. AULAWIN creates a corresponding sample table automatically, and the user can further specify sample dilution factors as well as sample weight and fill-up volumes for solid sample digestion.

The software makes it easy to measure a sample repeatedly. The result of each measurement is calculated automatically from the chosen calibration function and displayed in µg/l or µg/kg. The QC function assures a high level of reliability. A toolbar allows fast access to frequently used functions. The absorbance signal graph can be viewed in real time and may also be recalled later.

The analytical results are filed together with all data necessary for quality assurance (date, time, user ID, sample number, calibration data, method parameters, signal graph, and so on). Worksheet templates can be created and stored to minimize set-up time for routine work. The user can format report sheets so that

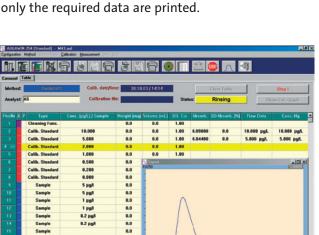


Abbildung: AULAWIN Probentabelle mit Meßsignal-Fenster (1 ppb-Peak)

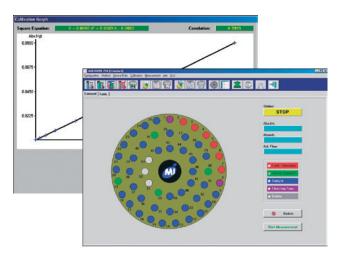


Figure: Calibration graph (back) and turntable screen (front)

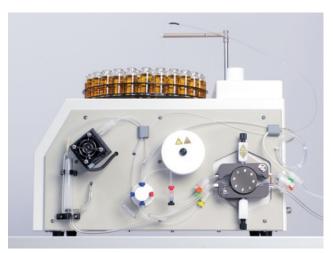


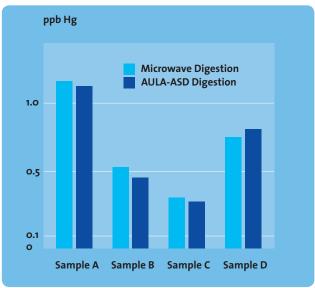
Figure: AULA-254 Gold reaction part

Extension:

Automated sample digestion (ASD) module

Samples frequently contain mercury in a complex form and require sample treatment prior to measurement. Wet chemical sample digestion turns out to be superior to thermal sample decomposition when water-based sample material is analyzed.

The AULA system is ready to be equipped with a sample preparation system for water samples and sample digestates. The ASD module carries out a digestion procedure derived from standard laboratory methods.



Comparison of microwave digested samples (light blue) and directly with AULA-ASD measured samples (dark blue)

Sample A: seepage water from chlor-alkali plant Sample B: tank rinsing water from chlor-alkali plant Sample C: effluents from PTFE production plant

Sample D: sewer plant effluent



The sample flow is continuously mixed with an oxidizing reagent (e.g. potassium permanganate, potassium dichromate). The sample/oxidant mixture is then heated to ca. 98°C in a reaction coil. As a result mercury is oxidised to Hg2+ and the sample matrix is destroyed by oxidative reactions. After the oxidation step hydroxylamine hydrochloride and tin-(II)-chloride are added to reduce the mercury to an elemental state.

Water samples measured directly with the AULA-ASD module (8 ml of sample + 2 ml HNO3 + 2 ml H2O2) yield results that closely match the same water samples digested with microwave. Automatic sample digestion is fast: cycle time for a complete analysis is less than 4 minutes.



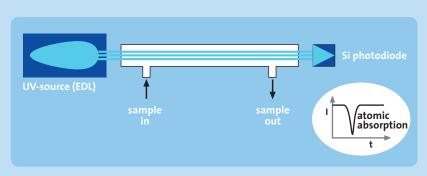
The AULA-ASD module is economical. Average reagent consumption for 100 analyses is: 3 g hydroxylamine hydrochloride, 1-5 g potassium permanganate, 6 g tin-(II)-chloride, 5 l water (deionized).

The AULA-ASD module is suited to any application where aqueous solutions require a sample treatment prior to analysis: surface water, ground water, seepage water, effluents, process water, and so on.

Cold Vapor Atomic Absorption Spectrometry: the ideal method for measuring mercury

The cold vapor atomic absorption technique is widely-used for mercury trace analysis because of its simplicity, robustness, and relative freedom from interferences.

For atomic absorption measurement it is important to know, that the atoms for the majority



of elements cannot exist in the free, ground state at room temperature. Therefore activation energy must be applied to the sample to break the bonds of molecules. A remarkable exception to this makes mercury. Free mercury atoms can exist at room temperature and, therefore, mercury can be measured by atomic absorption without heating the sample.

Excellent detection limits can be achieved with modern instruments like the AULA-254 Gold using preconcentration on a gold trap.

Standards for the AULA-System

Meets current regulations, with capacity in reserve to meet more stringent standards. Compliant with EPA Method 7470A (liquid waste), 7471A (solid or semisolid waste), 245.1 (drinking, surface, and saline waters, domestic and industrial wastes), 245.5 (soils, sediments, bottom deposits, and sludge type materials), 245.6, ASTM E538 (caustic soda and potash), FSIS

USDA Food Safety and Inspection Service Method for Mercury Determination in Food, ISO 16772 (soil quality), ISO 6637 (fruit, vegetables and derived products), ISO 11212-2 (starch and derived products), The Ontario Hydro Method (stack gas), European Method EN 1483 (water quality), EN 12497, EPA Method 245.7 (water), 1631 (water), EN 13806.

Technical Specifications AULA-254 Gold

Measuring principle:	Atomic Absorption, cold vapor technique (CVAAS)
Analytical wavelength:	253.65 nm
UV source:	Electrodeless low-pressure mercury discharge lamp (EDL)
Stabilization method:	Reference beam technique
Detector:	UV enhanced silicon photo diode
Optical cell:	Entirely made of fused silica, ca. 230 mm length
Stripping gas:	Argon, 4~6 l/h, stabilized with electronic mass flow controller (MFC)
Pre concentration principle:	Amalgamation on gold surface and subsequent release by quick heating
Gas-liquid separator:	Bubble-free, non-foaming crossflow principle design
Sample gas dehumidifier:	Thermoelectric principle (desiccant-free, low surface area)
Reagent / sample pump:	3-channel peristaltic pump, stabilized fixed speed (AULA -ASD: 6-channel pump
Autosampler:	53-place random access, carousel-type
Sample vials:	10 ml, glass; aluminium foil disc covers as accessory
Sample consumption:	ca. 1 ml – 3 ml
Heating coil temperature (AULA-ASD):	ca. 98 °C
Detection limit:	< 30 pg Hg (gold trap off); < 5 pg Hg (gold trap on)
Measuring range:	10 ng/L - 50 μg/L (gold trap off); 1 ng/L – 5 μg/L (gold tr ap on)
Zero drift:	Auto zero before each measurement
Measuring duration:	60 – 180 seconds typical
Software:	AULA-WIN, WindowsTM based
Electrical power supply:	115 V / 230 V~; 50 - 60 Hz, consumption ca. 100 W (AULA-254-ASD: 150 W)
Bench space requirements:	ca. 15"x17" WxD (37x44 cm), without PC
Weight:	approx. 40 lbs (18) kg, without PC



 $\stackrel{-}{\text{Manufactured according to ISO 9001 quality standard!}}$



The Response to an Analytical Challenge: Mercury Instruments.

Quantitative trace analysis of mercury has been a challenging task for the analyst until now. We from Mercury Instruments have made it our job to develop instruments for mercury analysis of the highest technical level. The range of applications for our mercury analyzers is unique world-wide.

More products by Mercury Instruments:

- Manually operated mercury analyzers
- Mercury vapor monitors
- Process analyzers for mercury
- Mercury immission monitors
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