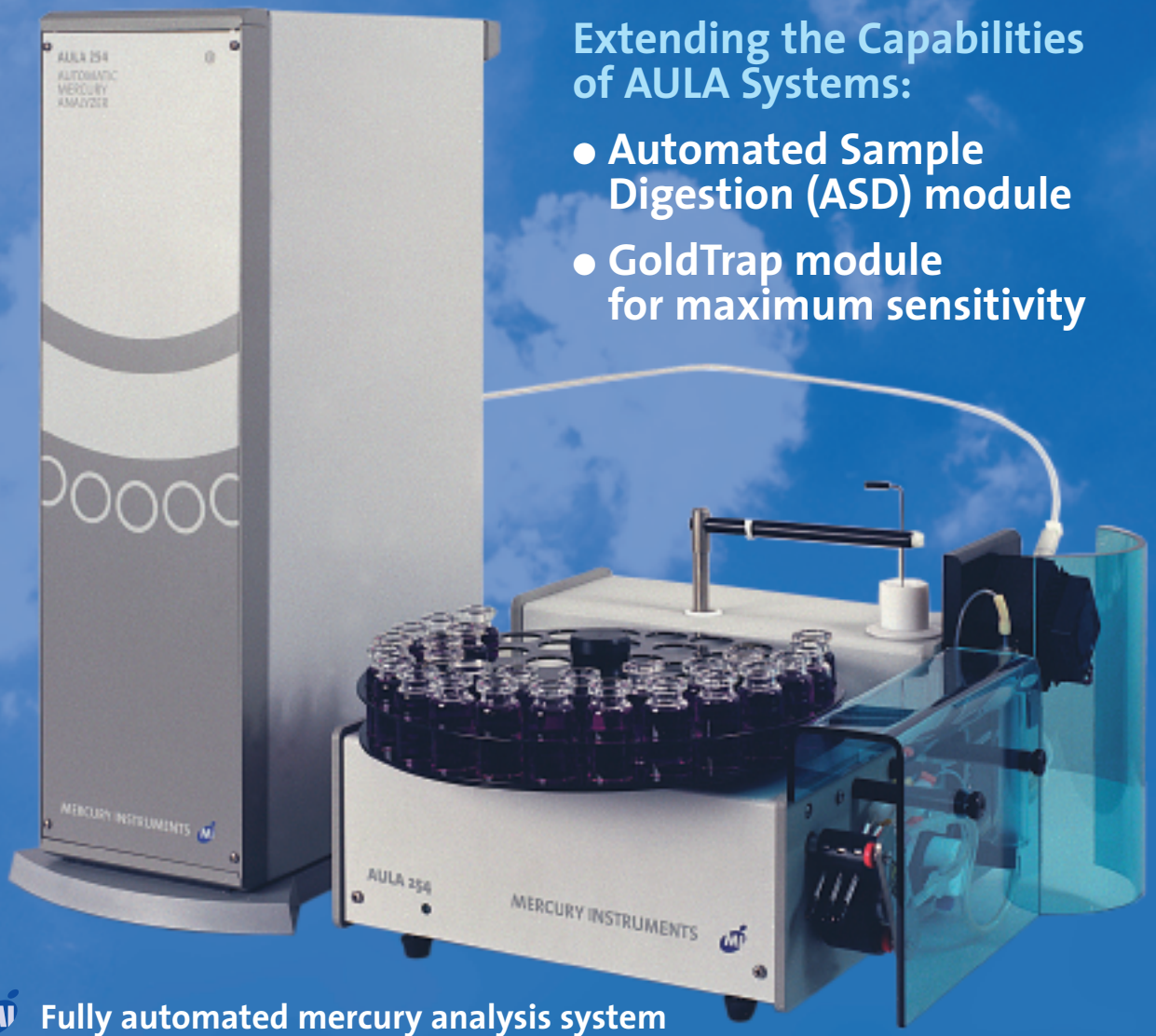
MERCURY INSTRUMENTS · ANALYTICAL TECHNOLOGIES

Automatic Mercury Analyzer
for the Laboratory

AULA 254
AULA Fluorescence



Extending the Capabilities of AULA Systems:

- **Automated Sample Digestion (ASD) module**
- **GoldTrap module for maximum sensitivity**

- **Fully automated mercury analysis system**
- **High performance flow analysis technique**
- **Two detection methods:**
  The AULA 254 uses Cold Vapor Atomic Absorption Spectrometry (CVAAS)
  The AULA Fluorescence uses Cold Vapor Atomic Fluorescence Spectrometry (CVAFS)
- **Widest dynamic linear range**
- **Robust modular construction**
- **AULAWIN software and PC included**

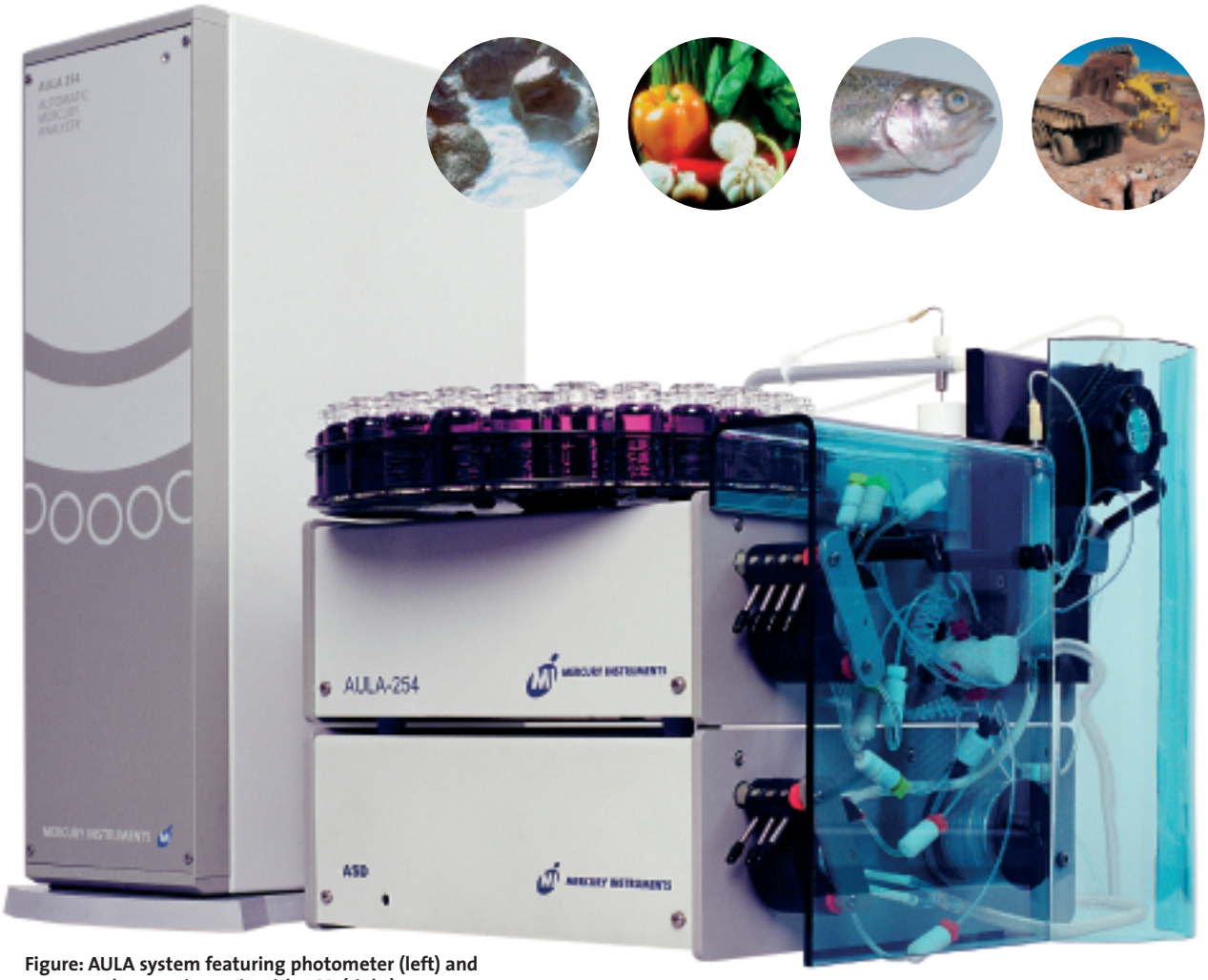


Figure: AULA system featuring photometer (left) and autosampler-reaction unit with ASD (right)

Applications

The AULA 254 and AULA Fluorescence are fully automatic analysis systems for determining trace mercury levels in a wide range of samples. The AULA systems are 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)**
- **Petrochemistry (oil products)**
- **Metallurgy and material testing**

Reliable and proven method

The system's working principle is the continuous flow technique. A continuous injection of stannous chloride in the sample flow transforms the mercury into elemental mercury. In a special crossflow reactor an argon stream strips out the elemental mercury and carries it into an optical cell. The mercury concentration is quantified in the cell, according to the AULA system being used:

I: the AULA 254 measures resonance absorption at a wavelength of 253.65 nm at ambient temperature. This analytical detection method is commonly known as cold vapor atomic absorption spectrometry (CVAAS).

II: the AULA Fluorescence quantifies resonance fluorescence at a wavelength of 253.65 nm at ambient temperature, a technique known as cold vapor atomic fluorescence spectrometry (CVAFS).

Optimized mercury detection technique

AULA 254: Atomic Absorption. 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 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 mercury analyzers.

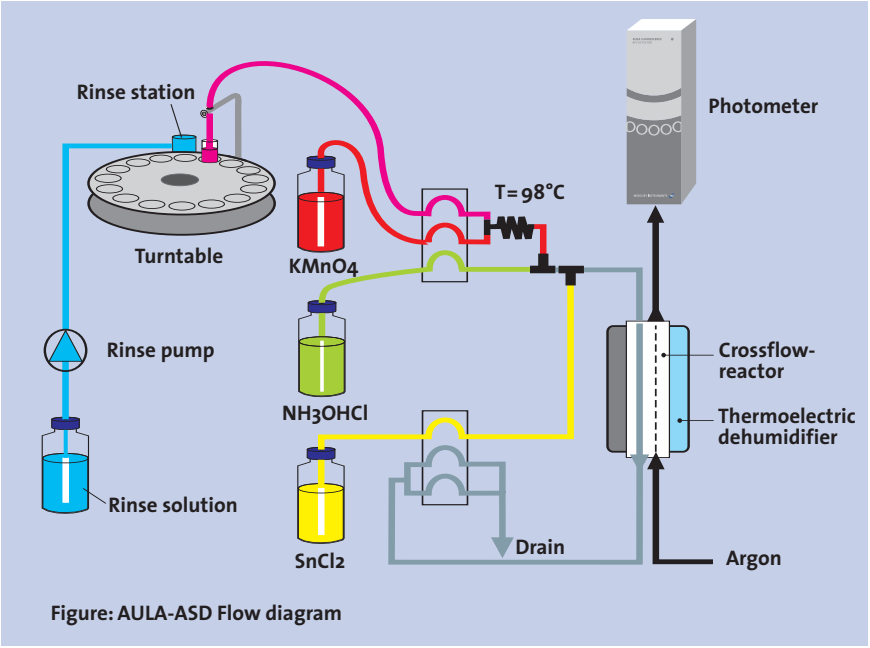


Figure: AULA-ASD Flow diagram

AULA Fluorescence: Atomic Fluorescence. The AULA Fluorescence employs the Channel Photomultiplier, a novel type of photomultiplier that can detect single photons. In combination with a very low background signal from our optimized low-reflection fluorescence cell, the AULA Fluorescence can reach extremely low (sub-ppt) detection limits.

High productivity

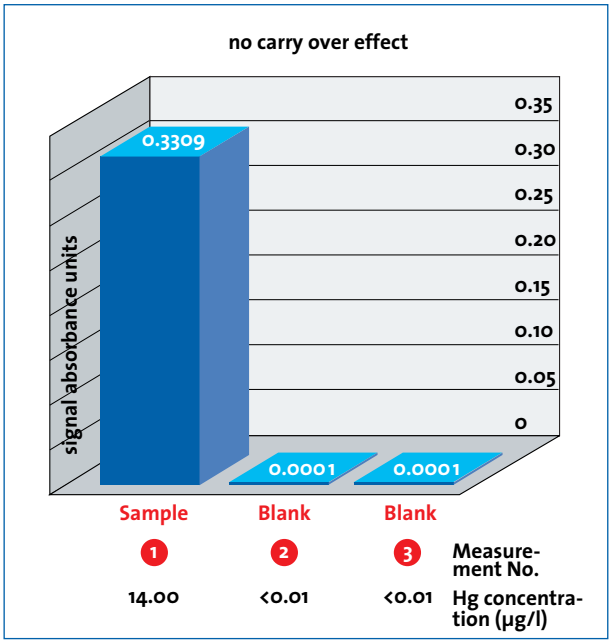
The typical duration of a full measurement cycle is 60 ... 180 seconds, depending on the set parameters. Measuring calibration standards, samples and QC standards is fully automatic. No long purging or rinsing procedure is needed, even when samples with high concentrations are analyzed.

The AULA system is simple to use. Samples are decanted into glass vials and positioned on the autosampler carousel. Reagent solution (tin-(II)-chloride) and rinse solution 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 re-measurement. New samples can be added at any time, even during a sample run.

Minimized memory effects

Mercury vapor tends to cling to surfaces, causing a carry-over (memory effect) that can compromise results. AULA systems minimize memory effect by using selected materials (FEP, borosilicate glass, Tygon LFL) 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, allowing a high sample throughput.



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.

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. In contrast to other dryers this device has an extremely small surface, which prevents mercury adsorption.

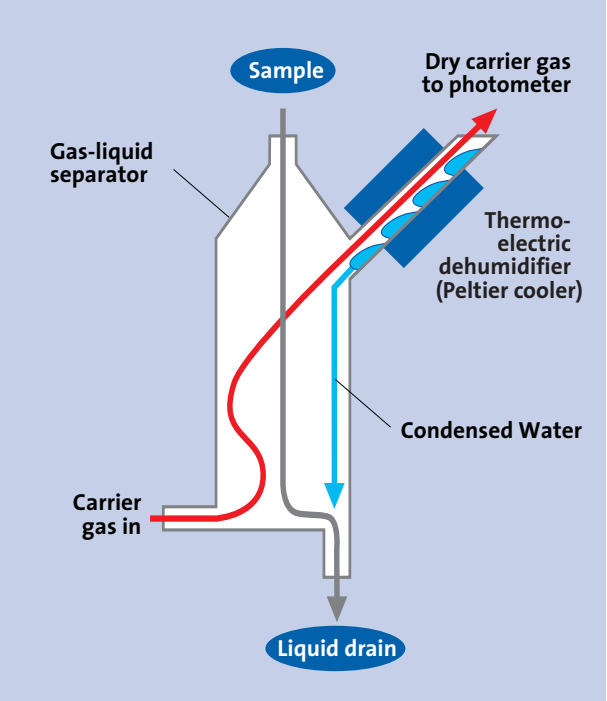


Figure: Schematic of gas-liquid separator with thermoelectric dehumidifier

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.

User safety

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.

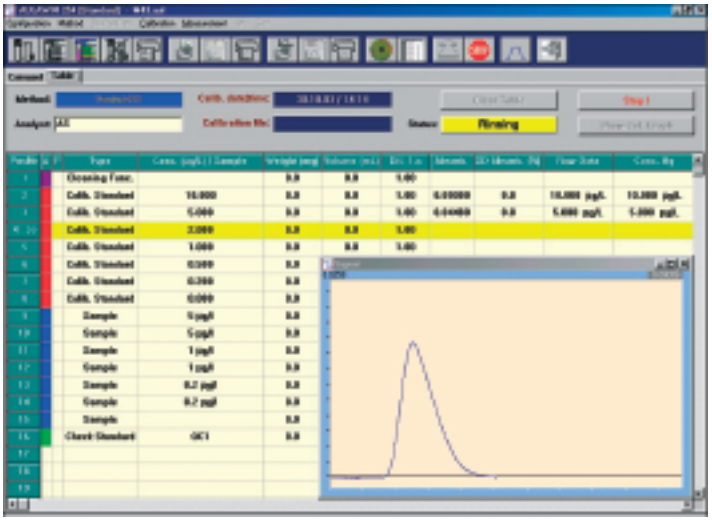
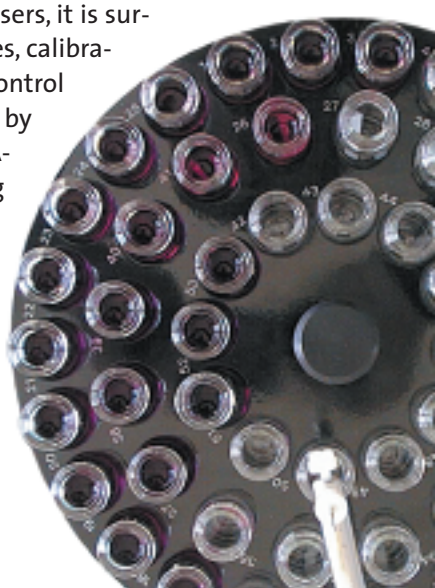


Figure: Screen print of sample table and measurement signal window (1 ppb)

Full-feature software

The AULAWIN software offers the complete feature set of modern analytical software. Developed with substantial input from our users, it is surprisingly 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.



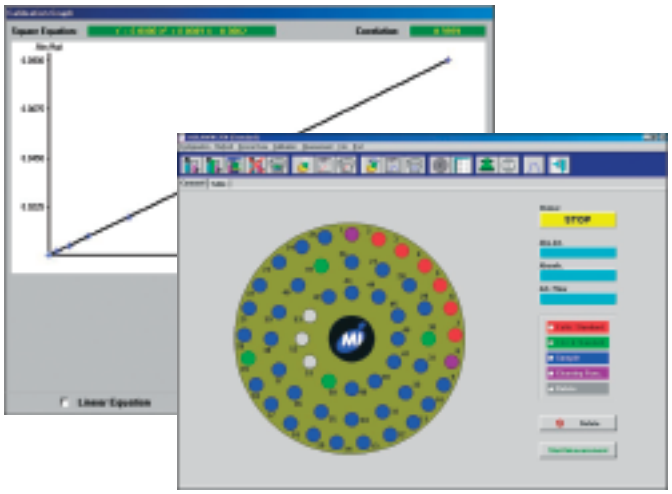
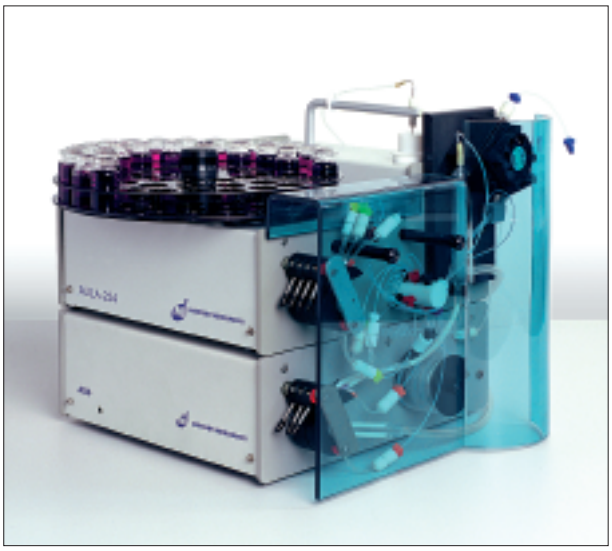


Figure: Calibration graph and turntable screen



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 only the required data are printed.

ACCESSORIES

Automated sample digestion (ASD) module

The AULA system can be equipped with a sample preparation system for aqueous samples. The ASD module carries out a digestion procedure derived from standard laboratory methods.

The module automatically draws sample from its vial and continuously mixes it 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. 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 HNO_3 + 2 ml H_2O_2) 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.

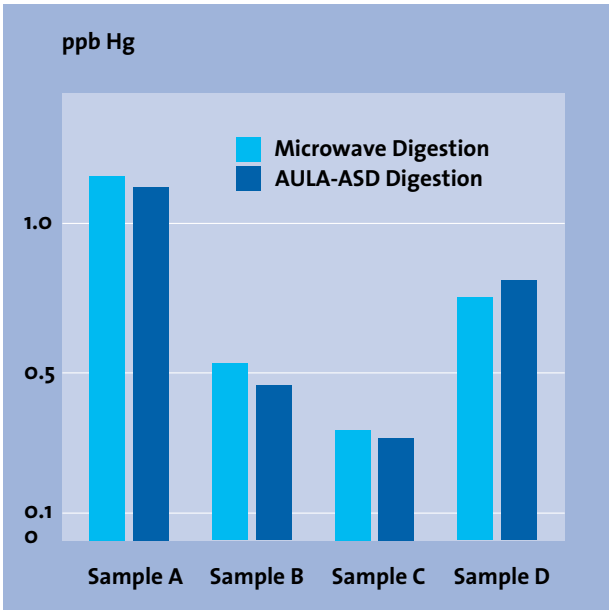


Figure: Comparison of microwave digested samples (light blue) and directly with AULA-ASD measured samples (dark blue)
Sample A: seepage water from chlor-alkali plant
Sample B: tank rinsing water from chlor-alkali plant
Sample C: effluents from PTFE production plant
Sample D: sewer plant effluent

GoldTrap module

The GoldTrap mercury preconcentration module drastically enhances detection sensitivity, to values lower than 1 ng/l. Using ultra pure reagents and following the guidelines for mercury analysis at ultra trace levels, the AULA 254 (using AAS) can achieve a measuring range from 1 ppt to 5000 ppt, and the AULA Fluorescence (using AFS) can achieve a measuring range from 0.05 ppt to 5000 ppt.

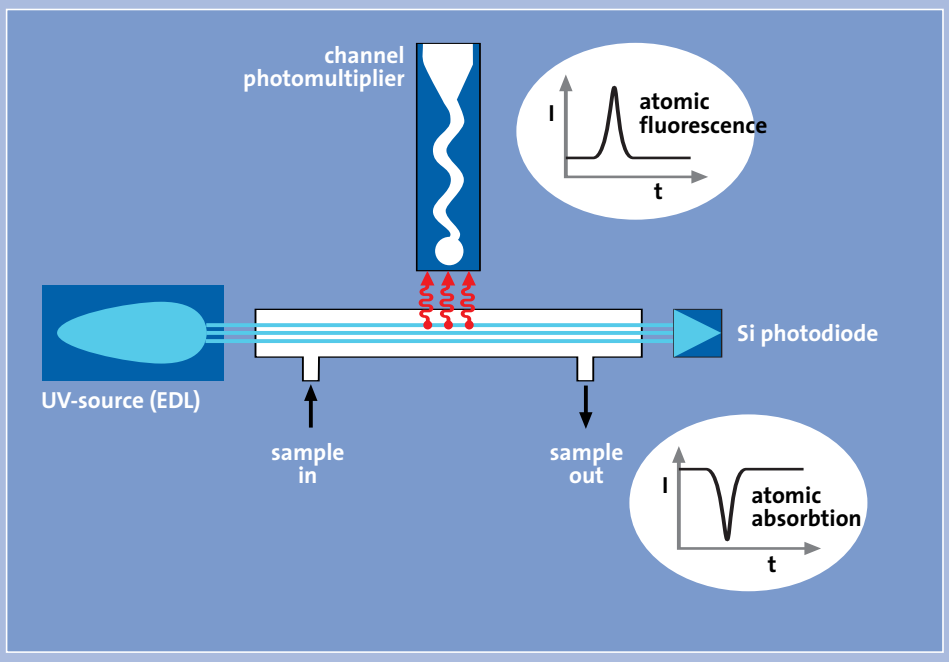
Small thermal inertia is an outstanding property of our latest GoldTrap design, achieved through the use of a wafer-thin ceramic substrate. Heating and cooling rates are now very fast. Analysis duration is comparable to the direct method.

The GoldTrap module 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.

Atomic Absorption versus Atomic Fluorescence: A Comparison

The cold vapor atomic absorption technique is commonly used for mercury trace analysis because of its simplicity, robustness, and relative freedom from interferences. Excellent detection limits can be achieved with modern instruments.

For even lower detection limits, users turn to the atomic fluorescence technique. However, the ultra-low limits can only be reached if extreme care is taken to keep blanks at lowest levels and contamination to a minimum. Precautions described in the appropriate standard methods must be strictly followed.



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), 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 [AULA 254 only] , EPA Method 245.7 (water), 1631 (water). EN 13806 and European Method EN 13506 (water) [AULA Fluorescence only]

Technical Specifications AULA 254 and AULA-Fluorescence

Measuring principle:	Atomic Absorption, cold vapor technique (CVAAS) Atomic Fluorescence, cold vapor technique (CVAFS)
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 Channel photomultiplier, photon count mode
Optical cell:	Entirely made of fused silica, ca. 230 mm length Low reflection fluorescence cell
Stripping gas:	Argon, 4~6 l/h, stabilized with electronic mass flow controller (MFC)
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: two pumps)
Autosampler:	53-place random access, carousel-type
Sample vials:	10 ml, glass; aluminium foil disc covers as accessory
Sample consumption:	ca. 1 ml
Heating coil temperature (AULA-ASD):	ca. 98 °C
Detection limit:	< 30 pg Hg < 3 pg Hg
Measuring range:	10 ng/L - 50 µg/L (10 ppt – 50 ppb) 0.5 ng/L – 10 µg/L (0.5 ppt – 10 ppb)
Zero drift:	Autozero before each measurement
Measuring duration:	60 – 180 seconds typical
Software:	AULA-WIN, Windows™ based
Electrical power supply:	115 V / 230 V~; 50 - 60 Hz, consumption ca. 100 W AULA with ASD module: 150 W
Dimensions:	Autosampler-reaction unit: 345 x 295 x 330 mm (WxHxD) Photometer: 160 x 450 x 360 mm
Bench space requirements:	ca. 90 x 70 cm (W x D), without PC
Weight:	approx. 14 kg total, without PC

Technical Specifications AULA 254 GoldTrap and AULA Fluorescence GoldTrap

Pre concentration principle:	Amalgamation on gold surface and subsequent release by quick heating
Detection limit:	< 5 pg Hg < 0.5 pg Hg
Measuring range:	1 ng/L – 5 µg/L (1 ppt – 5 ppb) 0.05 ng/L – 5 µg/L (0.05 ppt – 5 ppb)

Other specifications see AULA standard systems above.

Manufactured according to ISO 9001 quality standard!

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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.

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