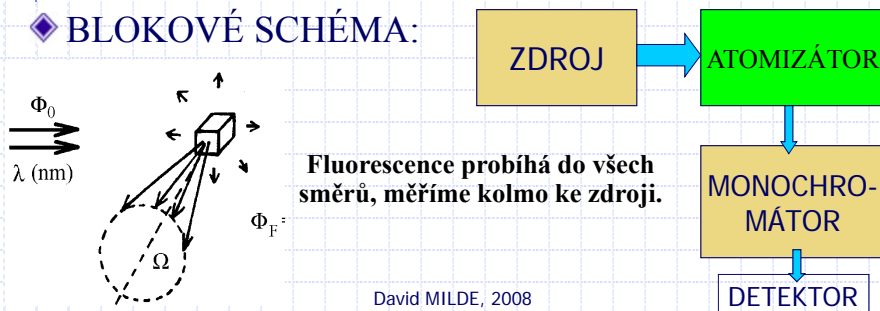


ATOMOVÁ FLUORESCENČNÍ SPEKTROMETRIE

Atomic Fluorescence Spectrometry AFS

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- ◆ Sleduje se emise záření plynnými atomy vznikající při přechodu e^- do nižšího energetického stavu, které byly excitovány absorpcí elektromagnetického záření z primárního zdroje (přechod e^- do vyššího energetického stavu).
- ◆ Fluorescence je proces s nízkou účinností (kvantová účinnost fluorescence $\Phi_F \approx 10^{-3}-10^{-4}$) \Rightarrow potřeba intenzivních zdrojů záření.
- ◆ AF spektrometr má analogické schéma jako AAS, fluorescenční záření se měří kolmo k budícímu zdroji.
- ◆ BLOKOVÉ SCHÉMA:



Atomová fluorescence

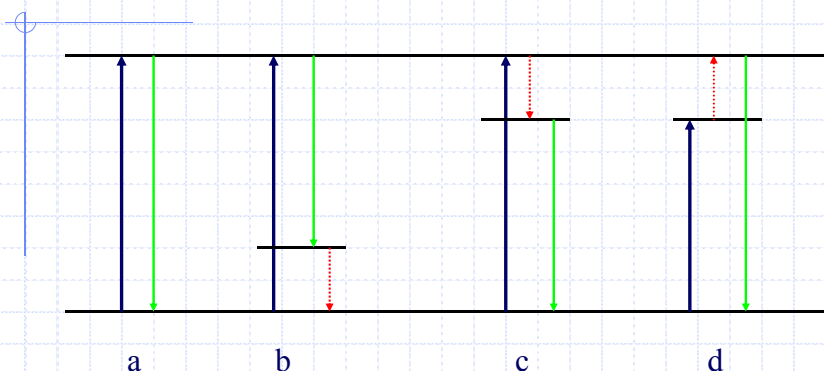
- ◆ Energetická výtěžnost fluorescence χ_F
- ◆ Kvantová účinnost fluorescence Φ_F

$$\chi_F = \frac{E_{emit}}{E_{abs}} \leq 1 \quad \Phi_F = \frac{N_{emit}}{N_{abs}}$$

- ◆ Zhášení fluorescence: kolize excitovaných atomů s ostatními částicemi atomizátoru – předání E bez vyzáření fotonu.
- ◆ Rozptyl fluorescenčního záření: na nevypařených částicích v atomizátoru a na optice spektrometru.

David MILDE, 2008

AFS – přechody elektronů



- a – rezonanční AFS b – přímá čárová fluorescence
c – postupná fluorescence d – termicky asistovaná fluorescence

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Součásti spektrometru

◆ PRIMÁRNÍ ZDROJE ZÁŘENÍ (měly by být intenzivní):

- Jako u AAS: výbojka s dutou katodou, bezelektrodová výbojka; EDL má vyšší intenzitu, ale není dostupná pro všechny prvky.
- Kontinuální zdroje (např. Xe lampa) – používají se zřídka, protože intenzita záření pro diskrétní λ je nízká.
- Lasery: LIF – laser induced fluorescence – monochromatické laserové záření je schopno vstupovat do interakce prakticky s jediným absorpčním přechodem a excitovat jedinou hladinu, zvýšení citlivosti stanovení, vysoká cena.

◆ DISPERZNÍ PRVKY:

- Bezdisperzní spektrometry – pro čárové zdroje; selektivita je dána zdrojem, který je schopen excitovat pouze atomy sledovaného prvku.
- Interferenční filtry nebo mřížkové monochromátory – pro kontinuální zdroje.

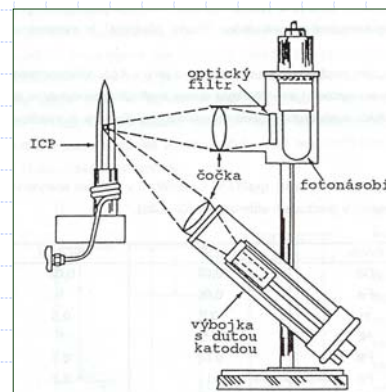
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Atomizační techniky v AFS

◆ Stejně jako u AAS:

- FA – plameny různého složení, kruhové hořáky.
- ETA – zejména ve spojení s LIF.
- HG, generování studených par pro Hg.

◆ ICP-AFS: indukčně vázané plazma:



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Výhody a nevýhody AFS

- ◆ **VÝHODY:** jednoduchá instrumentace; LOD lepší pro 10 prvků než u AAS (zejména ve vzdálené UV); velká linearita kalibrací; citlivost ovlivněna intenzitou excitačního zdroje.
- ◆ **NEVÝHODY:** rozptyl záření; zhášení fluorescence; samoabsorpce u vyšších koncentrací; LOD výrazně horší pro prvky s čarami ve VIS oblasti.
- ◆ **INTERFERENCE:**
 - spektrální – objevují se pouze u kontinuálních zdrojů,
 - nespektrální – závisí na atomizační technice.

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Analytical certainty down to the ng level

Increasingly stricter requirements

The growing industrialization, with the development of ever new technologies and materials, leads to increasing pollution of the environment.

Mercury is a particularly critical polluting element because of its toxicity. Accordingly, standards and legislation worldwide continue to become ever more stringent.

The maximum permissible concentrations of mercury are lower than those of any other routinely monitored element. High contents need to be determined with high certainty and repeatability down to the sub-nanogram level.

Modern techniques for mercury analysis have to meet a number of clear and exacting requirements:

- High sensitivity and selectivity
- High sample throughput
- Interference-free methods
- Easy routine operation

The method of choice: Atomic Fluorescence Spectrometry

The **mercur** analyzer detects mercury content by means of atomic fluorescence. Atomic fluorescence spectrometry, unlike absorption spectrometry, measures the fluorescence emitted by a sample. The intensity of the fluorescence emission is directly proportional to the intensity of the light source. To excite fluorescence in the sample, the **mercur** uses a high-energy (high-pressure mercury vapor lamp) with high light output adds considerably to the sensitivity of the method. Thanks to its high excellent detection limits and its wide linear measuring range, atomic fluorescence spectrometry is the method of choice in mercury trace analysis. With these advantages the working range of AFS can be expanded by about a decade towards both lower and higher concentrations.

An added advantage: Cold Vapor Technique

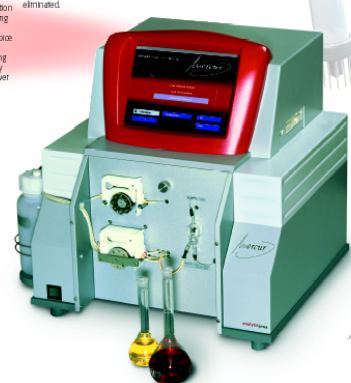
The **mercur** combines atomic fluorescence with the cold vapor technique, so you can make use of the advantages of both techniques. By the cold vapor technique, mercury is converted to the gaseous state by reduction of the dissolved cation with SnCl₂ and separated from the solution. The gaseous mercury is carried by an argon gas stream to the fluorescence cell.

Because the analyte is separated from the matrix, interferences and matrix effects are almost completely eliminated.

An expanding range of applications

Mercury analysis is employed in an impressive range of fields. Due to the harmful influence on the human organism, mercury is a focus of close attention. In medical disciplines such as occupational medicine and dentistry as well as in drinking water and food control.

The **mercur** has been designed for dependable mercury analysis in all these fields. Teamed with the customer support provided by Analytik Jena, the instrument permits the statutory concentration limits to be checked accurately and reliably.



Comprehensive data processing and quality control

Whether you use the **mercur** as a standalone instrument or as a PC-controlled system, its WinAPC control and data analysis software not only handles all everyday routine tasks but also meets the strictest requirements of quality control.


The software's automatic, versatile quality control system monitors your analytical data and releases appropriate instrument response if permissible limits are exceeded.

Increasingly stringent standards

Today's most stringent standards governing mercury determination in the USA and Europe are based on the atomic fluorescence method. The U.S. EPA method 1631 "Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry" requires a minimum level of quantitation of 0.2 ng/l and a detection limit of 0.2 ng/l (while specifying a maximum permissible concentration of 1.5 ng of mercury per liter in surface waters and non-polluted ground water. These figures make the EPA the world's most stringent statutory legislation on mercury.

Designed to meet these requirements, the **mercur** guarantees mercury analyses compliant with:

- EPA 245.7 and EPA 1631
- EN 12358



David MILDE, 2004

- ◆ P S Analytical offers many solutions to the determination of Hg, As, Se, Sb, Te and Bi. These instruments are mainly based on Atomic Fluorescence Spectrometry.

Why Atomic Fluorescence?

- ◆ Hydride forming elements and mercury all absorb and fluoresce in the UV region below 260nm where OES and AAS are relatively insensitive.
- ◆ High intensity excitation sources available.
- ◆ Low spectral interference-analyte separated from matrix by the hydride/vapour generation process.
- ◆ High selectivity: only the analyte corresponding to the excitation source will fluoresce.
- ◆ Excellent sensitivity and linearity.



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AI 3300 Atomic Fluorescence Spectrometer (Aurora Instruments Ltd., Canada)



Element	Detection Limit (µg/L)	RSD (%)
As, Se, Pb, Bi, Sb, Te, Sn	0.01	<1.0
Hg, Cd	0.001	<1.0
Zn	<1.0	<1.0
Ge	<0.05	<1.0

Linear Range Over 10⁵

Specifications

Sample Atomization

Atomizer: Quartz tube furnace with automatic ignition of Ar-H₂ diffusion flame reduces interference
 Furnace Heating: Computer controlled heating
 Carrier Gas: Argon

Sample Preparation & Delivery

Vapor/hydride Generator: Continuous flow, peristaltic pump, high performance mixing section, gas-liquid separator for cold-vapor mercury determinations and hydride generation determinations of As, Se, Te, Bi, Sb, Sn, and other hydride forming elements
 Peristaltic Pumps: A 6-channel pump with 2 adjustable pressure control clamps and programmable speed control
 Exhaust System: Exhaust system with filter efficiently decontaminates pollutants
 Gas/Liquid Separator: High efficiency, two-stage gas/liquid separator design

Optics

Optics Design: Short focal length, non-dispersive, integrated optical design
 Dual Channel: Dual channel for simultaneous two-element analysis, using computer controlled, modulated, pulsed light sources
 Light Sources: Specially-designed high intensity hollow Cathode Lamps (HCL) with 2 channel independent power supply, providing improved sensitivities and lower detection limits.
 Detector: High quantum efficiency, solar blind photomultiplier tube (PMT).

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