



food analysis



commodities analysis



pharma analysis

## GALAB Laboratories

Your partner for  
analytical services

environment analysis



# Mercury Speciation in Routine Analysis

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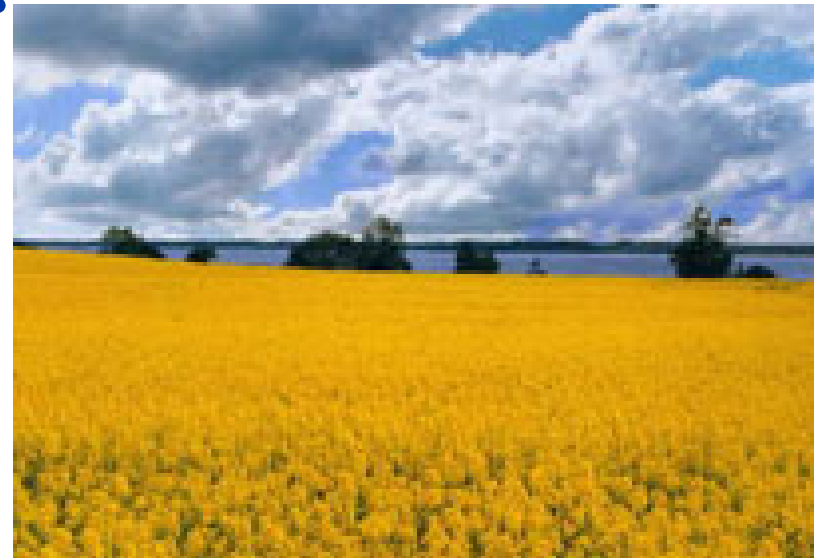
# GALAB Laboratories

- first contract laboratory performing “speciation analysis”
- founded in 1992
- located in Hamburg, Germany
- 30 employees
- accredited I7025
- certified acc. DIN ISO 9001



# environmental monitoring

- elemental species (Hg, Sn, As)
- persistent organic pollutants
- endocrine disruptors
- pharmaceuticals
- pesticides



# food analysis

- pesticides
  - ca. 400 GC-MS
  - ca. 200 LC-MS/MS
- pharmaceuticals
  - ca. 200 LC-MS/MS
- elements
- other contaminates



# materials testing

- residue monitoring
- quality testing



# pharmaceutical analysis

- stability testing
- GLP, GMP testing
- method validation



# definition of routine analysis

The objective of routine analysis is to achieve a defined and homogeneous level of precision and accuracy of analytical results over a **long period of time**





# way to routine analysis

- phase I: development of a new method
- phase II: preparation of routine analysis
- phase III: internal quality assurance
- phase IV: external quality assurance



# phase I

- preparation for first calibration
- test of linearity
- working range
- precision
- matrix influences
- optimization

=> publication of results



# phase II

- preparation of routine
- writing SOP
- lab staff training
- robustness tests
- establishing of QA data
- documentation



# phase III

- internal QA
  - keep the method running
  - comparability
  - personnel
  - lab organization
  - lab facilities
  - document control



=> accreditation



# phase IV

- external QA
  - inter lab comparison
  - auditing

=> continuous improvement



# elements of method validation

- specificity
  - tests for interference, matrix influence and artifacts
- precision
  - repeatability, intermediate precision, reproducibility
- accuracy
  - recovery tests, CRM
- detection limit, quantitation limit
  - statistically estimated according ISO norms
- robustness
  - personnel variation, parameter variation
- linearity, working range



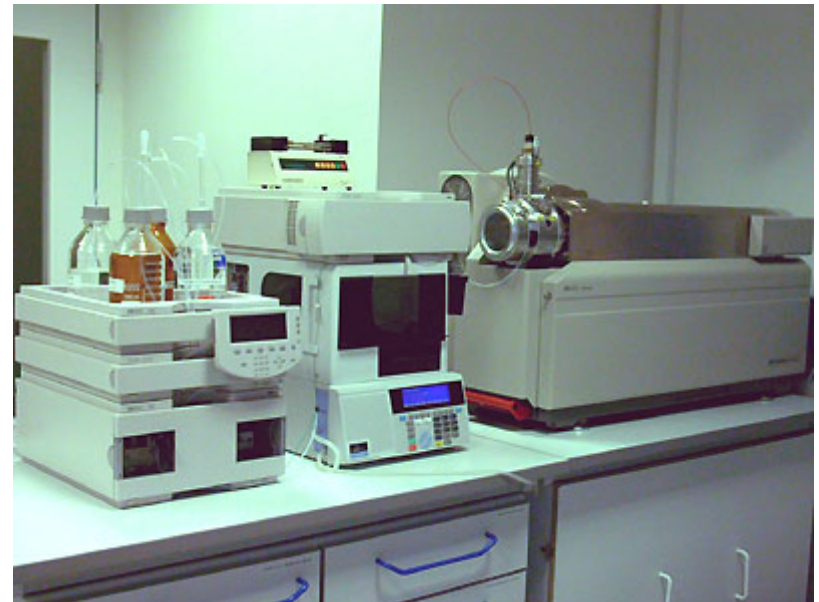
# Applicability of methods

Method	Species	Research	Routine
CV-AAS	Hg <sub>tot</sub> , Hg(0)	--	++
CV-AFS	Hg <sub>tot</sub> , Hg(0)	o	+
HPLC-AAS	Hg <sub>org</sub>	o+	--
HPLC-AFS	Hg <sub>org</sub>	o+	--
GC-ICP-MS	MeHg, Me <sub>2</sub> Hg	+	o
GC-AED	MeHg, Me <sub>2</sub> Hg	+	o+
GC-AFS	MeHg, Me <sub>2</sub> Hg	+	o+
HPLC-ICP-MS	Hg <sub>org</sub>	++	-o
IC-ICP-MS	Hg(II), Hg <sub>org</sub>	++	-o



# tasks of routine Hg analysis

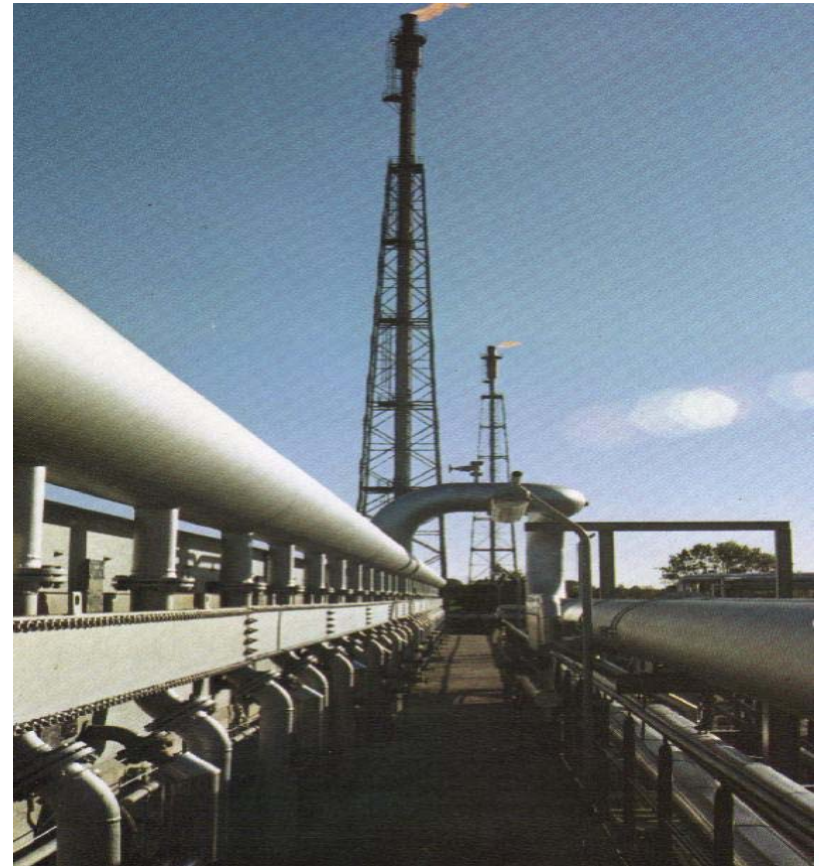
- environmental monitoring
  - water
  - sediment
  - air
- contaminated sites
- working place
- food
- corrosion





# Hg in natural gas

- corrosion of Al heat exchangers
- specification 10 ng Hg/m<sup>3</sup>
- high pressure sampling
- Hg condensation
- iso kinetic sampling
- off-line vs. on-line sampling



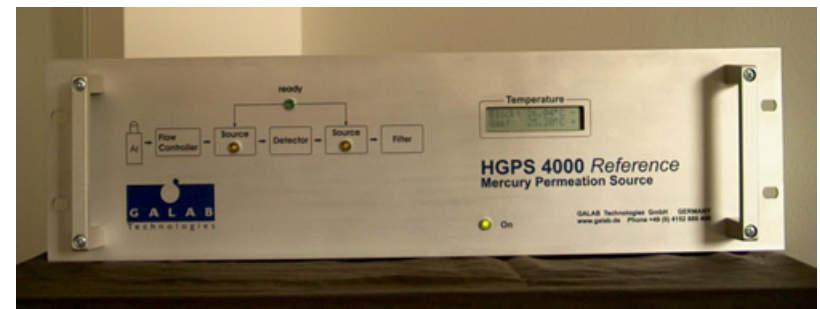
# Hg(0) on-line measurement

- automated on-line instrument for the determination of Hg in gas
  - hydrogen
  - natural gas
- LOD 1 ng Hg/m<sup>3</sup>
- range 1-100 ng Hg/m<sup>3</sup>
- low maintenance
- PC with RS232 controller



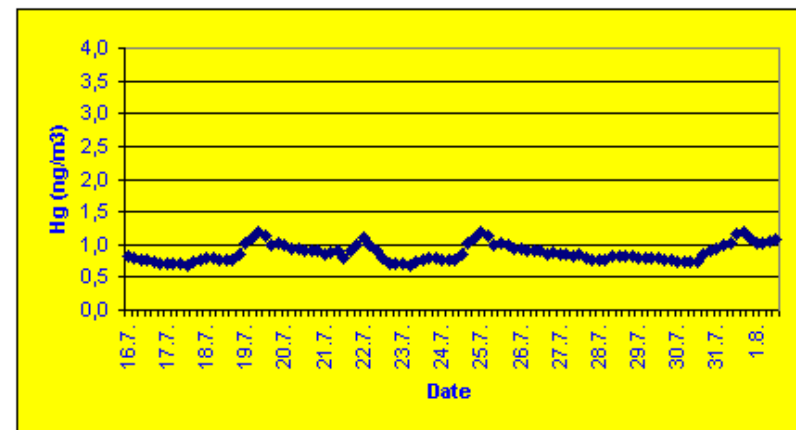
# Hg(0) perm source

- auto calibration unit
- Hg(0) perm source
- thermostated to 24 °C
- mass flow controller



# Hg(0) online monitoring

- sampling rate 20 –60 min
- monitoring of Hg in a hydrogen pipeline at DOW chemical plant



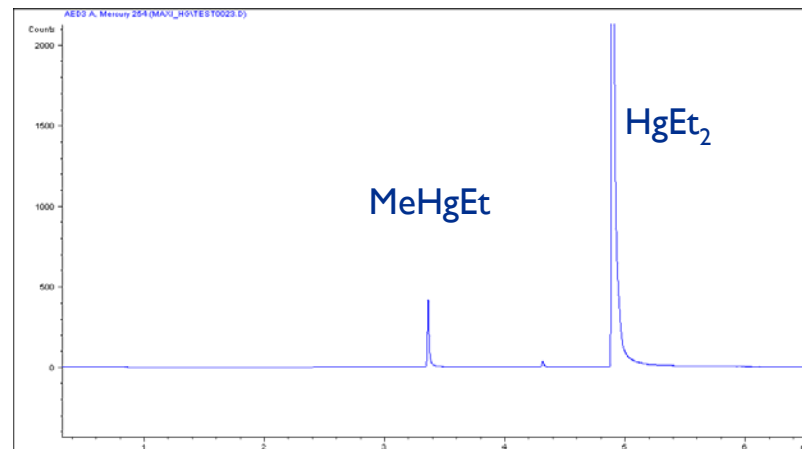
# off-line sampling

- sputtered gold traps
- low Hg blank
- DIN-ISO conform
- ambient air monitoring
- working place monitoring
- contaminated site
- working range 1-50 ng/m<sup>3</sup>
- applicable to CV-AFS



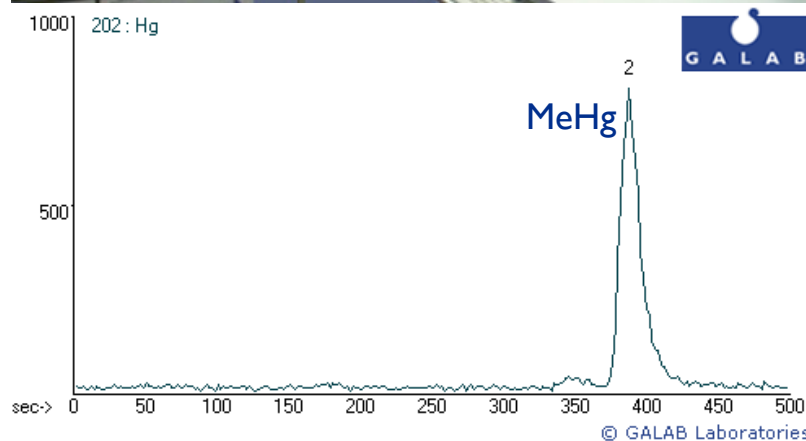
# GC-AED

- MeHg
- extraction followed by derivatization
- ethylation  $\text{NaBEt}_4$
- propylation  $\text{NaBPr}_4$



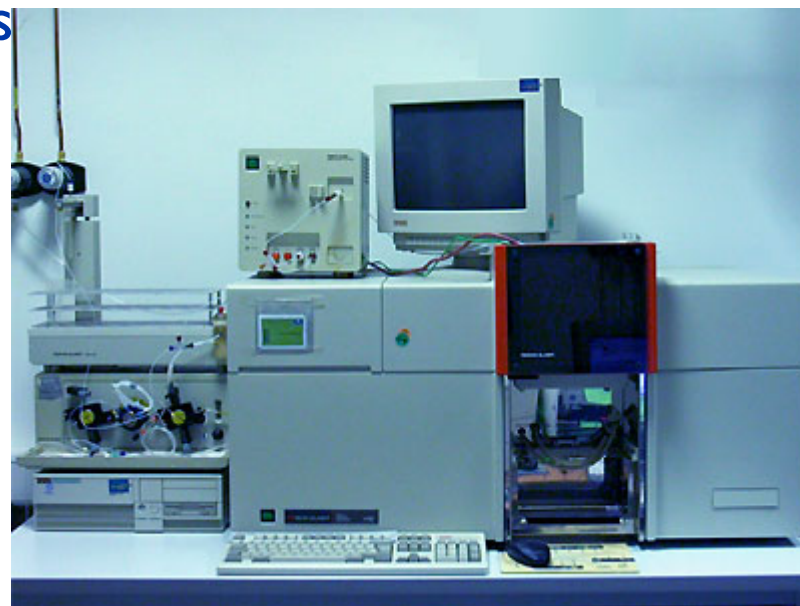
# IC-ICP-MS

- ion chromatography hyphenated with ICP-MS
- Hg(inorg), MeHg, Hg(org)
- sample preparation without derivatization
- “expensive”



# flow injection AAS

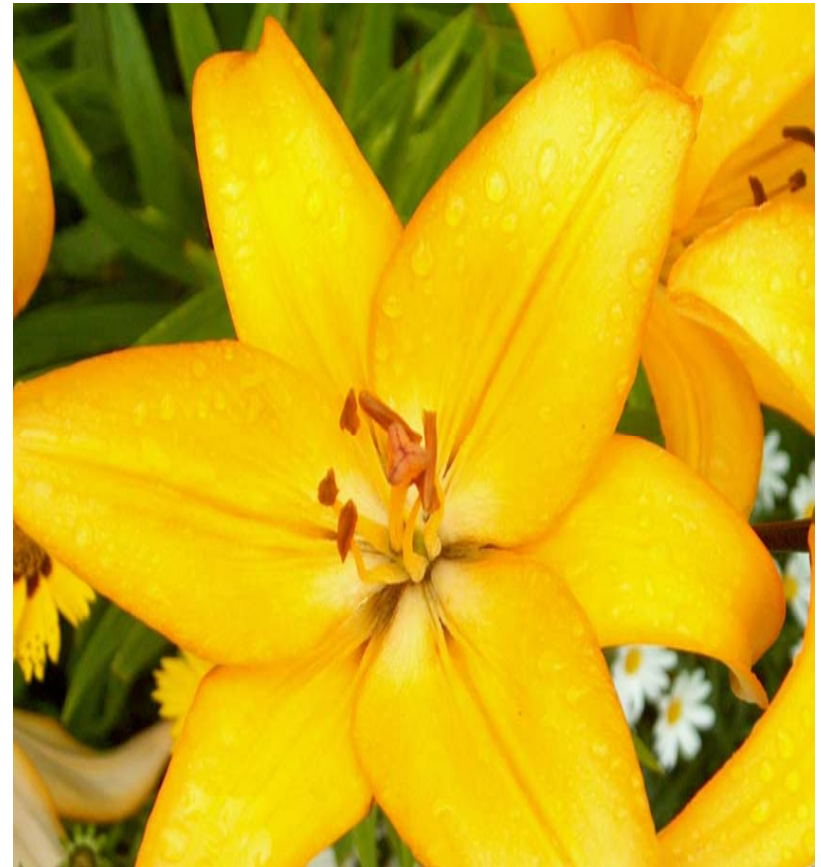
- Hg(tot)
- acid digestion of the samples
- robust
- routine instrument
- “cheap”





# summary

- routine usually begins when research ends
- objectives for routine analysis are
  - monitoring the quality
  - monitoring limit values
- traceability of data
- data may be defeated in court



# thank you for your attention!

## acknowledgment

Wolfgang Frech  
Michael Sperling

