

## Bulk and Trace Analysis

### Technologies:

- *X-Ray Fluorescence Spectrometry, XRF*
- *Atomic Emission Spectrometry by Inductively Coupled Plasma, ICP-AES*
- *Carrier Gas Heat Extraction, CGHE*
- *Carbon-Sulfur-Analyzer, CS*
- *Mass Spectrometry by Inductively Coupled Plasma, ICP-MS*

### Equipment:

- *XRF*: Pioneer, Bruker-AXS
- *ICP-AES*: OPTIMA 4800 DV, Perkin-Elmer
- *ICP-MS*: 7500 ce, Agilent
- *CGHE*: TC 600, LECO
- *CS*: CS 600, LECO

## Laser Ablation - Inductively Coupled Plasma - Mass Spectrometry (LA-ICP-MS)

### Technology:

*Mass Spectrometry of Material Ablated by Laser, Surface and Micro Area Analysis*

### Equipment:

- *ICP-MS*: 7500 ce, Agilent
- *UP 193 FX*, New Wave

## Auger Electron Spectroscopy (AES)

### Technology:

*Nano Area Analysis  
Surface and Interface Analysis*

### Equipment:

*Auger Nanoprobe PHI 680, Physical Electronics*

### Category:

**C. Particle Characterisation ex-situ**

### Institute:

**KIT**

### Location:

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## Short technology description/Overview and Main Features (Equipment Capabilities) of Bulk and Trace Analysis:

- **XRF**: After excitation with high-energy X-rays the sample emits "secondary" (or fluorescent) X-rays. The spectrometry of these characteristic X-rays gives qualitative (B – U) and precise quantitative (0,01 – 100%) information.
- **ICP-AES**: Liquid or dissolved samples are sprayed in an inductively coupled Ar Plasma, where the excited atoms emit light with characteristic wavelength. The simultaneous spectrometer provide qualitative (all elements except H, O, N, C, halogens, noble gas) and quantitative (0,001 – 100 %) information.
- **ICP-MS**: Liquid or dissolved samples are sprayed in an inductively coupled Ar Plasma, where atoms are ionized and accelerated into a quadrupol massspectrometer. Mass interferences can be eliminated by an octopole reaction cell. All elements from Li to U can be analysed (0,000001 – 100 %), except C, N, O, F, Cl and noble gases because of the liquid and atmospheric background. Isotopes can be determined.
- **CGHE** (gas fusion analysis): Under He carrier gas flow the sample is heated with a metal flux to max. 2500 °C in a graphite

crucible. Oxides are reduced to CO and CO<sub>2</sub> and determined by infrared absorption in a gas cell (0,0001 – 50%).

- **CS:** The sample is inductively heated in flowing oxygen with a metal as combustion and heating aid. The formed CO<sub>2</sub> is detected by non dispersive IR-absorption (0,0001 – 100%)

### Short technology description/Overview and Main Features (Equipment Capabilities) of LA-ICP-MS:

LA-ICP-MS is a sensitive analytical method (0,000001 – 100%) for rapid multi-element (Li - U except C, N, O, F, Cl) determination in the trace and ultratrace range of different solid sample materials, technical products and even biological samples. A small area (Ø 10 – 200 µm) of the sample is vaporized in a laser plasma by focused laser radiation and transported with helium and argon into the inductively coupled plasma ion source of an ICP-MS. There the material is atomized, ionized, accelerated into the mass spectrometer and separated according the mass/charge ratio and energy/charge ratio and detected by electron multipliers.

The main advantage of LA-ICP-MS is that samples are investigated not under vacuum but in atmospheric pressure and electrically conducting as well as non conducting material can be investigated.

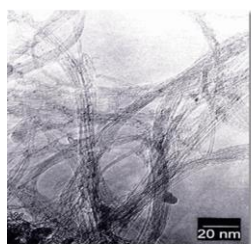
### Short technology description / Overview and Main Features (Equipment Capabilities) of Auger Electron Spectroscopy:

Auger Electron Spectrometry (AES) provides information about composition and to some extent chemical state within nanometer size of solid and vacuum-stable, non-insulating materials of rough, multilayer and fracture surfaces. In combination with Ar-ion-sputtering depth profiles to 1000 nm are available.

- Semi-quantitative analysis of Li to U; quantitative analysis with standards
- practical detection limit 0,1 to 1 at-%
- Multi point and area analysis, linescans, element maps
- Resolutions: practical Auger electron analysis < 20 nm, depth analysis 0,5 – 5 nm depending on Auger electron energy
- Two types of ion guns available: scanning (1x1 mm) ion spot (0,1 mm) and low energy ion gun
- Zalar Rotation™ for better interface resolution

In-situ fracture of samples with liquid N<sub>2</sub> cooling for grain boundary analysis

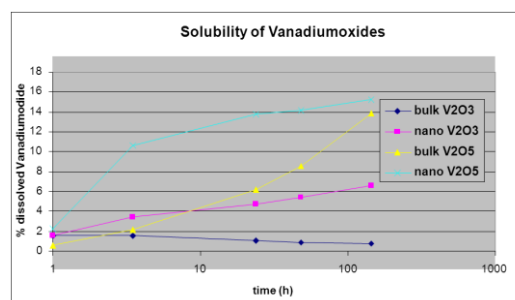
### Typical Samples & Images of Bulk and Trace Analysis:



<b>C</b>	<b>73 %</b>	<b>Ca</b>	<b>0,03 %</b>
<b>O</b>	<b>9 %</b>	<b>Fe</b>	<b>0,02 %</b>
<b>N</b>	<b>0,7 %</b>	<b>Ti</b>	<b>0,02 %</b>
<b>Mo</b>	<b>6,1 %</b>	<b>W</b>	<b>0,01 %</b>
<b>Co</b>	<b>1,6 %</b>	<b>Al</b>	<b>0,004 %</b>
<b>B</b>	<b>0,6 %</b>	<b>Cr</b>	<b>0,003 %</b>
<b>Mg</b>	<b>0,09 %</b>	<b>Cu</b>	<b>0,003 %</b>

ICP-OES, CGHE, CS

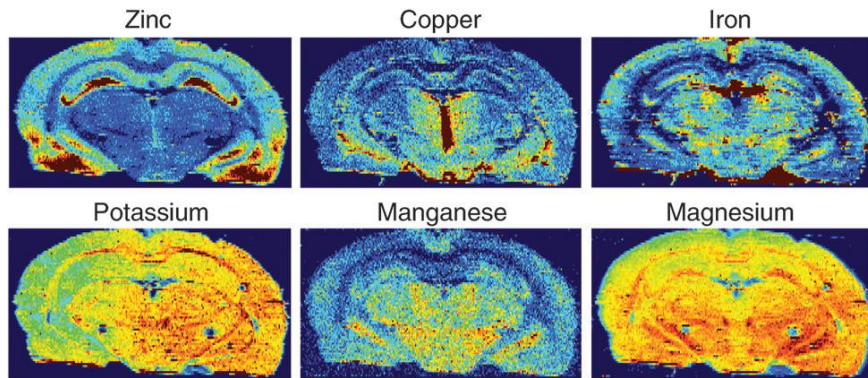
Analysis of Carbon nanotubes



Solubility of Vanadiumoxides in Lung Cells: ICP-AES, -MS

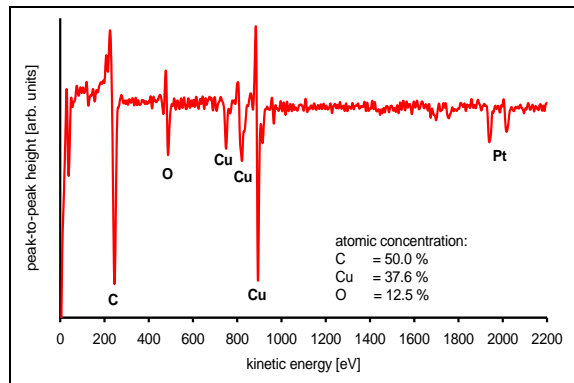
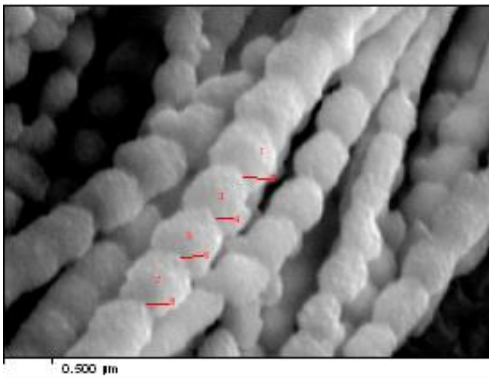
### Typical Samples & Images of LA-ICP-MS:

As LA-ICP-MS is i.e. successfully used in bioimaging of metals in brain tissue (S. J. Becker; FZ-Jülich) this method can be applied to investigate the interaction of inorganic nanopowder with cells.



Images of selected elements in a rat brain section by LA-ICP-MS (from *Metallomics*, 2010, 2, 104f, Sabine Becker et al.)

**Typical Samples & Images of Auger Electron Spectroscopy:**



SEM image of Cu nanowire and AES point analysis

Any further Information: