

For the chemical characterization of the bulk material for micro and nano materials five different analytical instruments are operated for the KNMF by the analytical group of the Institute of Applied Materials - Applied Material Physics (IAM-AWP):

- **X-Ray Fluorescence Spectrometry, XRF** (S4 Pioneer, Bruker-AXS)
- **Atomic Emission Spectrometry by Inductively Coupled Plasma, ICP-AES** (OPTIMA 4300 DV, Perkin-Elmer)
- **Mass Spectrometry by Inductively Coupled Plasma, ICP-MS** (7500ce, Agilent)
- **Carrier Gas Heat Extraction, CGHE** (TC 600, LECO)
- **Carbon-Sulfur-Analyzer** (CS 600, LECO)

## Contact

See KNMF website or contact the KNMF User Office.

## Equipment

### X-Ray Fluorescence Spectrometry, XRF (S4 Pioneer, Bruker-AXS)

#### Specification

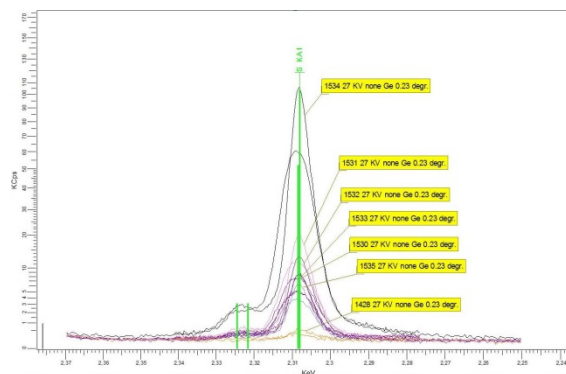
- Sequential wavelength dispersive X-ray spectrometer
- Detectable elements: (B) F to U in the concentration range from ppm to 100%
- Non-destructive analysis for qualitative and semi-quantitative determinations
- Sample forms like powder, solid, paste, film, liquid with size of 10 to 50000  $\mu\text{m}$
- Precise quantitative determination of samples prepared in fused borate beads or with polished surface

#### Accessories

Grinding, pelletizing, fusion machines

#### Typical samples

Precise determination of main compounds like Si, Ti, Al and other minor elements in nano powders of  $\text{SiO}_2$ ,  $\text{TiO}_2$ ,  $\text{Al}_2\text{O}_3$ , glass, other nano oxides/carbides/nitrides



**Fig. 1:**  
*S K $\alpha$ 1. Analysis from a filter cake on a paper filter.  
Concentration range 0.01 – 3.0 mass %.*

# Bulk and Trace Analysis of Nanomaterials



## Atomic Emission Spectrometry by Inductively Coupled Plasma, ICP-AES (OPTIMA 4300 DV, Perkin-Elmer)

### Specification

- Echelle grating optical system combined with prisms and two segmented charge coupled device (SCD) detectors enables simultaneous measurement of all elements except noble gas, halogens, hydrogen, oxygen and nitrogen
- Element concentrations ranging from below 1 µg/g (depending on sensitivity) to 50% in solids and < 0.001 to 100 mg/L in liquids

### Accessories

For solid samples all kind of dissolution techniques, i.e. microwave assisted digestion

### Typical samples

Widely used, one of the most versatile methods of inorganic and organic analysis: liquids, electrolytes, dissolved solids of metals, oxides, nitrides, carbides

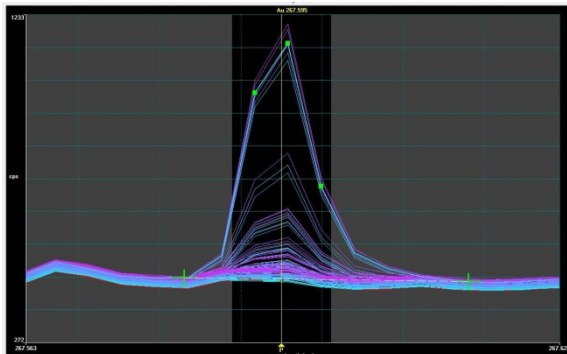


Fig. 2: Au with the ICP-OES by 242.795 nm. Chemical digestion from a mixture of Au and TiO<sub>2</sub> NM with aqua regia. Concentration range 0.005 – 0.20 mg/l

## Mass Spectrometry by Inductively Coupled Plasma, ICP-MS (7500ce, Agilent)

### Specification

- Quadrupole mass spectrometer with off-axis Omega lenses and Octopole
- Reaction System (ORS) to eliminate polyatomic interferences
- Mass range: 6–260 amu, He–U
- Ultra trace analysis ranging from below 1 ng/g (depending on sensitivity) to 1000 µg/g in solids and < 0.001 to 100 µg/L in liquids

### Accessories

For ultra-trace analysis: sub boiling point distillation, laminar flow bench

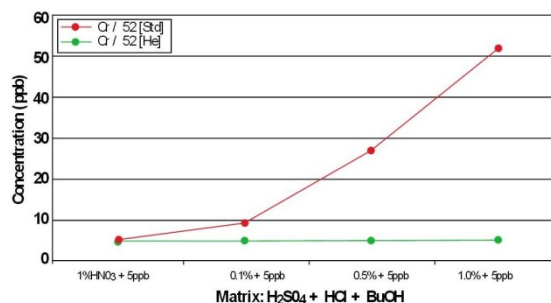


Fig. 3: Consistent interference reduction in a variable, complex matrix using He mode. Comparison plots showing Std mode (no cell gas - red) and He mode (green) spike recovery data for 5 ppb Cr in a variable matrix (up to 1% each of HCl, H<sub>2</sub>, SO<sub>4</sub> and Butanol). Potential interferences on 52 Cr include ArC, ClOH and SO.

# Bulk and Trace Analysis of Nanomaterials



## Carrier Gas Heat Extraction, CGHE (TC 600, LECO)

### Specification

- Simultaneous Nitrogen and Oxygen determination using IR and thermal conductivity by melting the sample in a graphite crucible in a metal bath at 2600°C with He as carrier gas
- Analysis range: < 0.00001 to 50%

### Typical samples

Solids: metals, inorganic materials like oxides, nitrides etc.

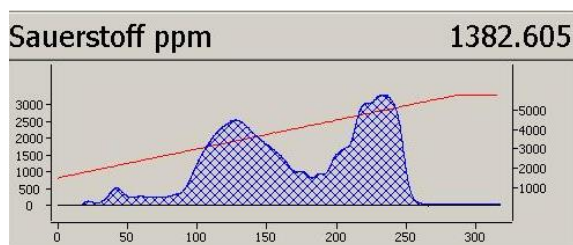


Fig. 4:  
Different kind of oxygen bonds in steel

## Carbon-Sulfur-Analyzer (CS 600, LECO)

### Specification

- Simultaneous Carbon and Sulfur determination by combustion in a high frequency furnace in oxygen flow using IR-detection of CO<sub>2</sub> and SO<sub>2</sub>
- Analysis range: < 0.0005 to 100%

### Typical samples

Solids: metals, inorganic and organic materials

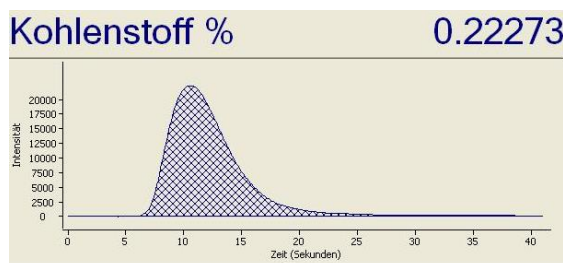


Fig. 5:  
C in WC