Analytical methods in BGR for research and exploration of ore deposits/mining residues

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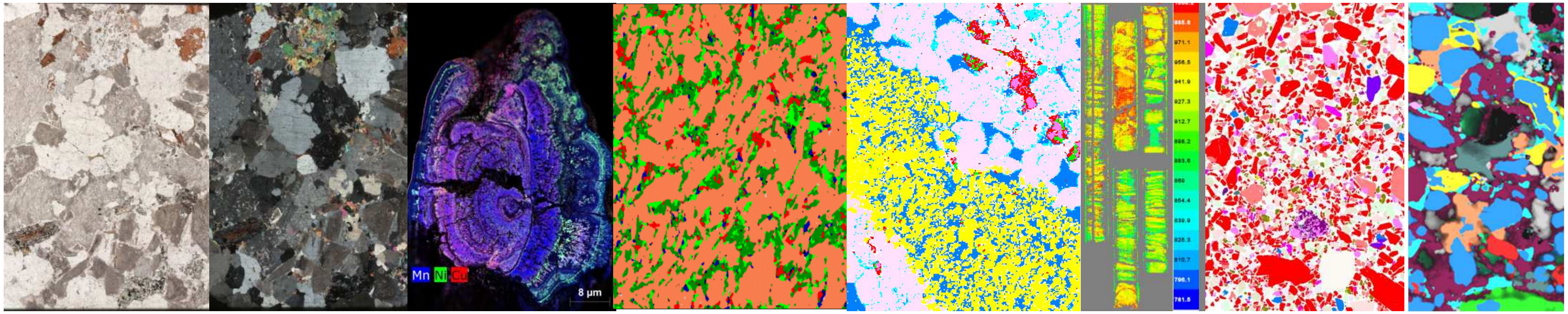
B1.2 „Mineral Residues“
Hannover, Germany

Optical // N X N EDXRF Classified LIBS (class.) Hypersp. REM_MLA Raman



OUTLINE

- BGR
- Existing cooparation with Chile
- Analytical Infrastructure of BGR
- Focus: scanning methods
- Case study: weathering of As-rich mining residues
- Conclusions

Federal Institute for Geosciences and Natural Ressources (BGR)

BGR is the central geoscientific authority providing advice to the German Federal Government in all geo-relevant questions.
It is subordinate to the Federal Ministry for Economic Affairs and Energy (BMWi).

In the field of Raw Materials Research, BGR investigates and assesses mineral potentials. Some major aspects are:

- Potential of ore deposits of strategically relevant elements
- Potential for efficiency improvement in processing
- Development of exploration strategies, exploration methods
- Development of innovative analysis strategies/methods for characterisation of ore samples and mining residues.

To achieve these goals, and to secure the supply of mineral resources, BGR cooperates with institutions, universities and industry.

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[1] Joint administration of BGR and LBEG in accordance with the administrative agreement concerning the formation of a Geological Survey of the Federal Republic of Germany dated Nov. 17/26, 1958
[2] Permanent post at LBEG
[3] Organisationally appointed to Z.8
[4] provisional
[5] entrusted with management of affairs

Existing cooperation with Chile (1/2)

- BMBF CLIENT I (2013 - 2016)
- BMBF CLIENT II (waiting for approval)
- Bilateral cooperation BGR-SERNAGEOMIN for scientific evaluation of mine tailings (2016 – 2018)
  - Investigation of tailings for possible valuable and toxic elements.
  - First field trip in May 2016: Drilling campaign at three processing sites in region IV (around Ovalle).
    - 19 drilling holes (max.8m)
    - 132 drilling meter

Delirio

Ovalle

Existing cooperation with Chile (2/2)

- Bilateral cooperation BGR-SERNAGEOMIN (cont.)
  - More field work is planned in June/July 2017
  - Locations: ENAMI Taltal, Las Luzes, Planta Paposo
  - Additional drilling by BGR
  - In-situ XRF measurements (Cu, Fe, Pb, Zn, As) by Fugro Consult
  - Elaboration of 3D tailings models with metal distribution

Delirio

Delirio

Analytical infrastructure of BGR

- *Chemical lab for routine analysis of bulk samples*
    - XRD, XRF, ICP-MS, ICP-OES, etc.
- *Micro-analytical lab*
    - Laser ablation – ICPMS
    - Microprobe
    - Optical microscopy (transmitted and reflected light, CL)
    - SEM with EDX– MLA (automated mineral liberation analyzer)
    - Raman microscope
    - LIBS scanner and microscope
    - EDXRF-microscope for mapping
    - Hyperspectral core scanner
- *Field equipment*
    - Hyperspectral scanner, XRF handheld, LIBS+RAMAN handheld (2017)
    - Geophysical instruments (Radar, electrical resistivity, SIP etc.)

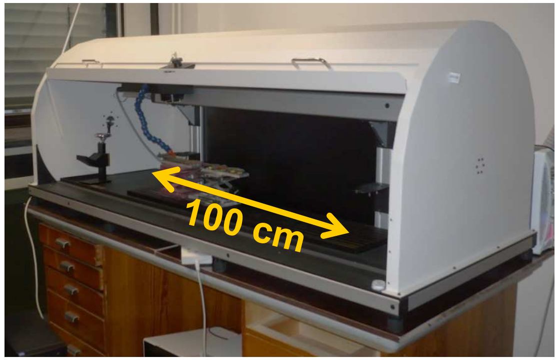
Why scanning methods?

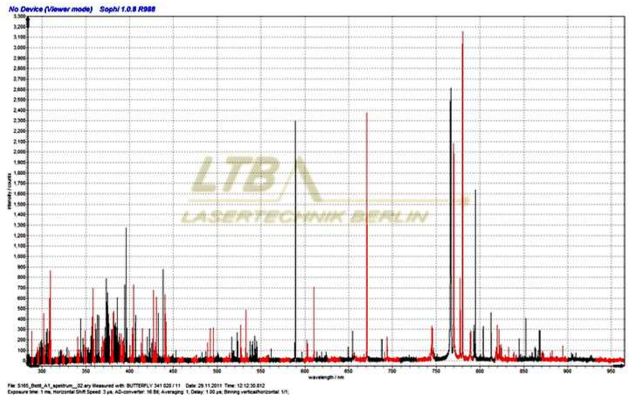
- Evaluation of the economic potential of deposits requires extremely high amounts of geochemical/mineralogical data.
- Real-time analyses for process control during production
- Standard procedures accepted by the industry are accurate, but time consuming, cost and personnel intensive.
- Scanning methods may provide most of the data in shorter time, at sufficient accuracy and information level, but at lower costs.

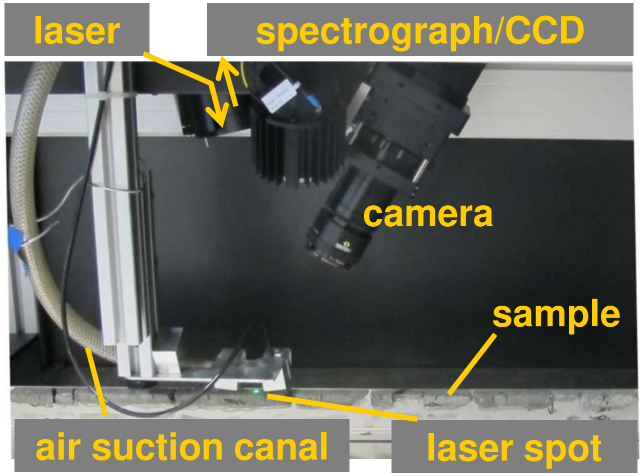
LIBS scanner – measurement principle

LIBS: **L**aser-**I**nduced **B**reakdown **S**pectroscopy

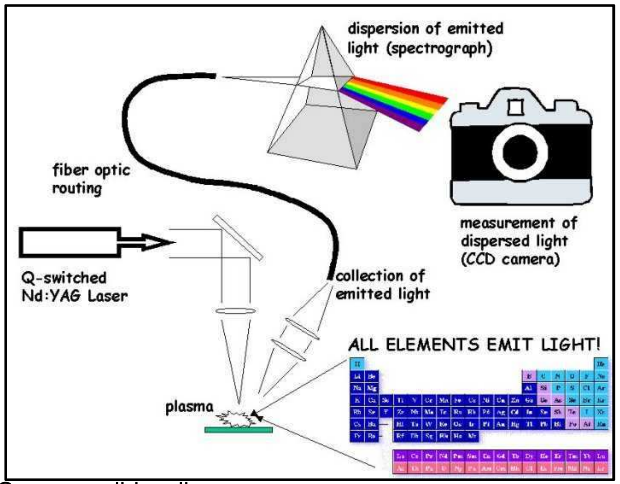
based on atom emission spectroscopy







laser	Nd:YAG 1064nm 50 mJ; 20 Hz
laser spot size	~200 µm
detector system	Echelle spectro-graph + CCD 285-945 nm (res. 28-94 pm)

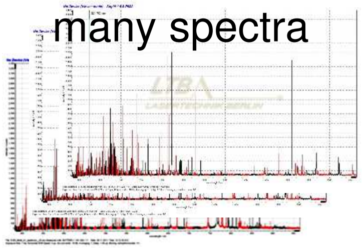


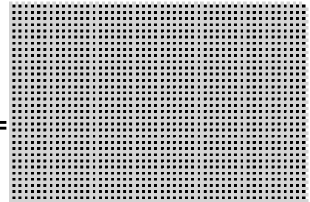
Source: wikipedia

LIBS data processing (1/3)
raw data

7m drilling core (tailings)
1D-profiling

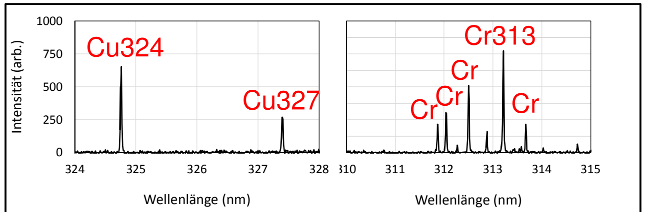
many spectra



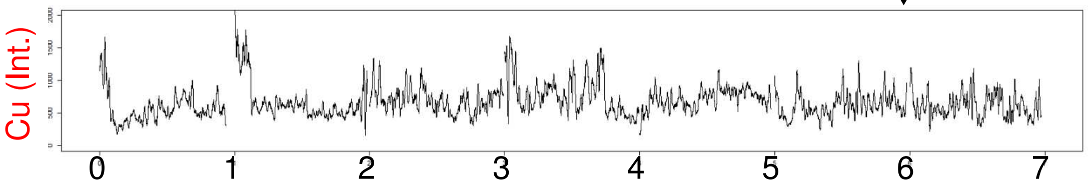


sample section

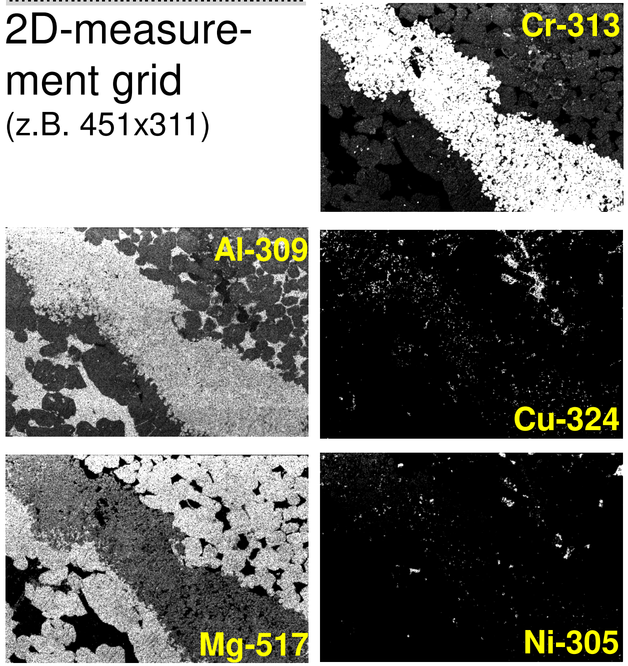
2D-measure-ment grid
(z.B. 451x311)



integr. of individual emission lines

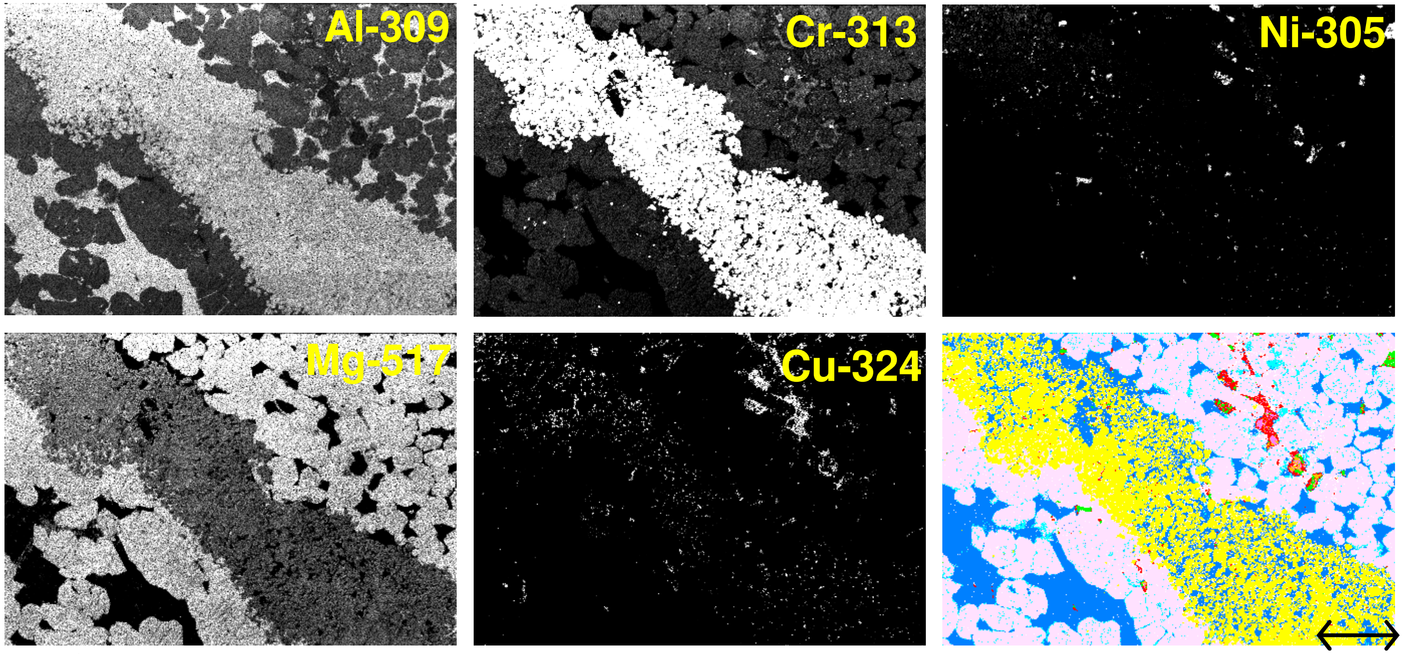


Element profile for Cu in tailings
resolution 200 µm



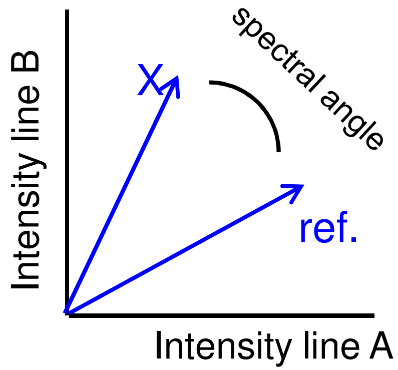
element maps (res. 60 µm)

Data Processing (2/3)
classification into mineral phases



LIBS microscope: 451x311 spectra, resolution 60 µm 5 mm

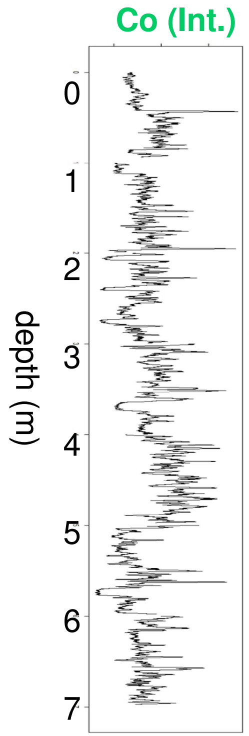
SAM: Spectral Angle Mapper



(source:ENVI-tutorial)

chromite	pentlandite	pyrrh./pyrite	diopside (pyroxene)
chalcopyrite	pyrite	plagioclase	o-pyroxene
			unknown

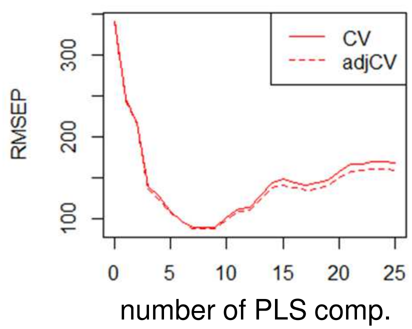
LIBS data processing (3/3) quantification

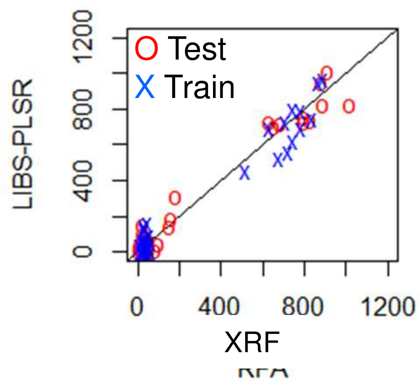


raw data

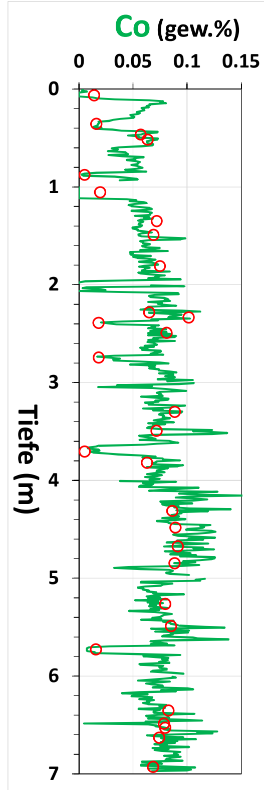
PLSR: **P**artial **L**east **S**quare **R**egression

- Finds correlation by projecting spectral and reference data to a new space
- PLS-comp with „scores“ und „loadings“
- Regression with only few PLS-comp.





σ(Co) 85 ppm
LOD 290 ppm



calibrated data

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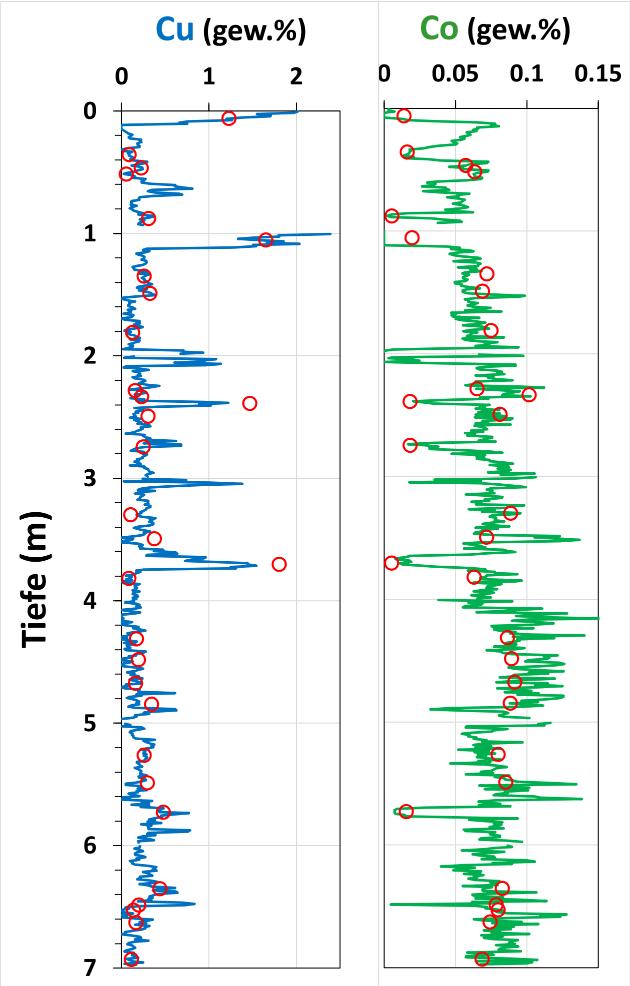
Example copper tailings (Chile)



LIBS drilling core analysis

	Cu (%)	Co(%)
MW (RFA)	0.28	0.076
MW (LIBS)	0.29	0.068
σ (LIBS)	0.08-0.15	0.007-0.008

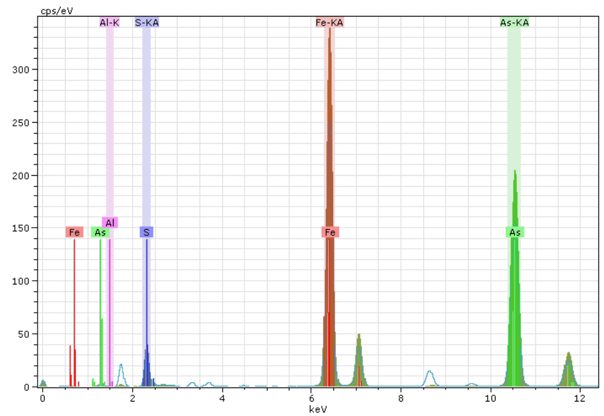
EDXRF microscope

EDXRF: Energy Dispersive X-ray Fluorescence

- Rhodium-tube (50 kV, 600 µA)
- beam diameter 17µm
- two detectors facing each other at 180°
- evacuation sample chamber (z ≥ Na)
- plane surface for samples of up to 20x15 cm
- measurement time: few ms pro spectrum (mapping 10^6 spectra in 30 min)

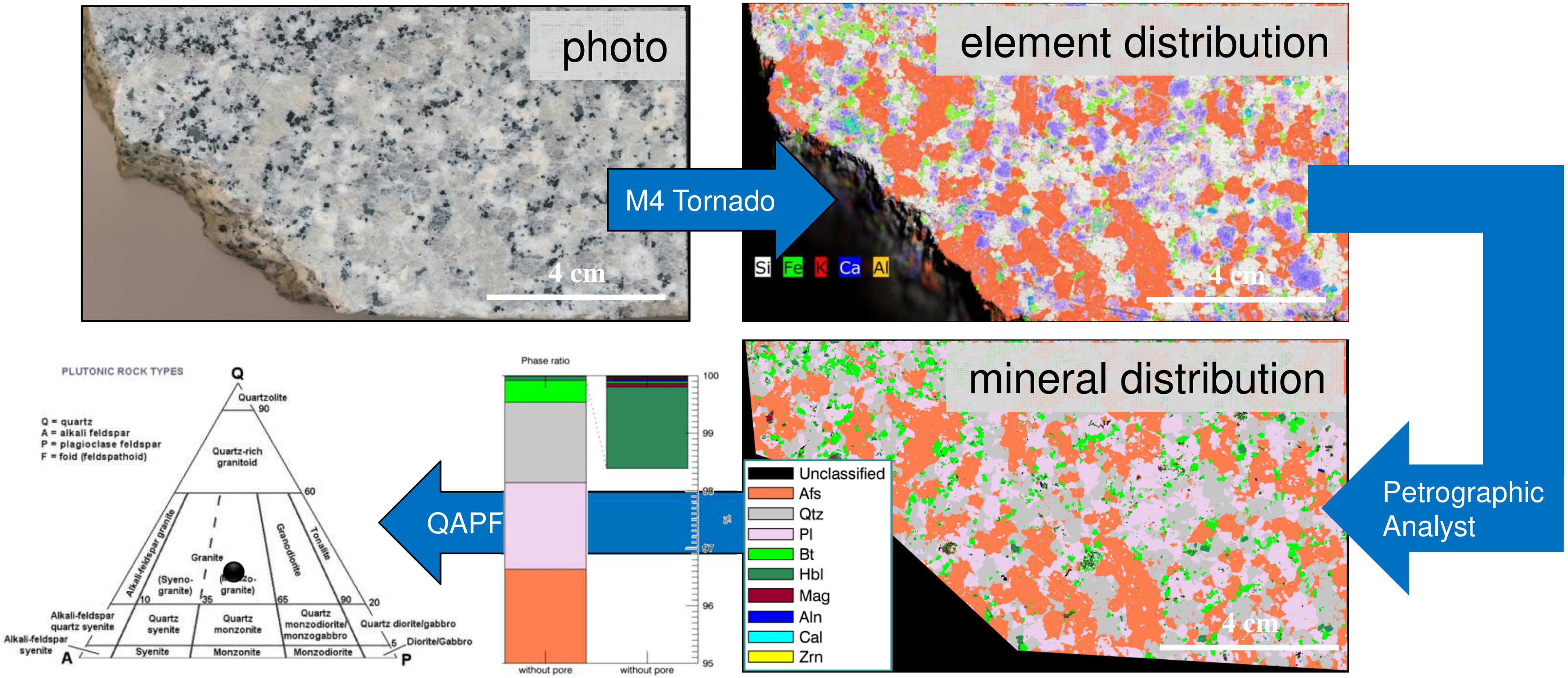
µ-EDXRF M4 Tornado

Element	AN	norm. C [wt.%]	Atom C [at.%]
Fe	26	32.74	31.99
As	33	47.77	34.79
S	16	19.36	32.94
Al	13	0.13	0.27
Total		100%	100%



EDXRF measurement of arsenopyrite mineral grain

EDXRF - semi-automatic data processing



Textural evaluation with
image analysis/Petrographic Analyst →

- veins
- grain size
- orientation
- paragenesis
- porosity
- rock nomenclature

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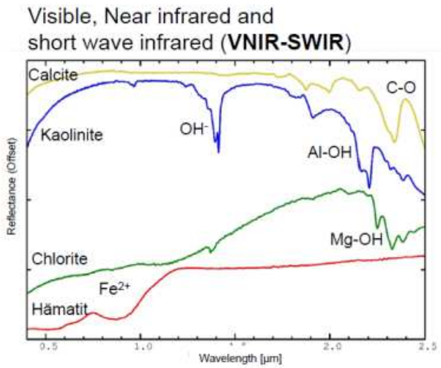
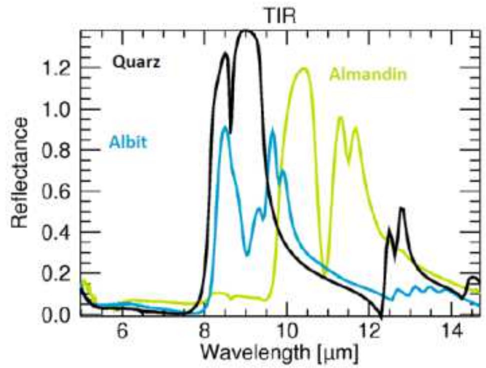
Source: W. Nikonov (BGR)

Hyperspectral imaging (1/2)

- Combines digital imaging and spectroscopy
- Each pixel contains a continuous spectrum
- Allows fast identification of various minerals





Core scanner: three line cameras, various objectives, motorized stage 65 x 120 cm

Sensor	Wavelength (nm)	Pixel size (µm)	Pixel
VNIR	400 – 1000	25 - 133	1000
SWIR	1000 – 2500	25 – 400	384
LWIR	7600 – 12500	100 – 400	350

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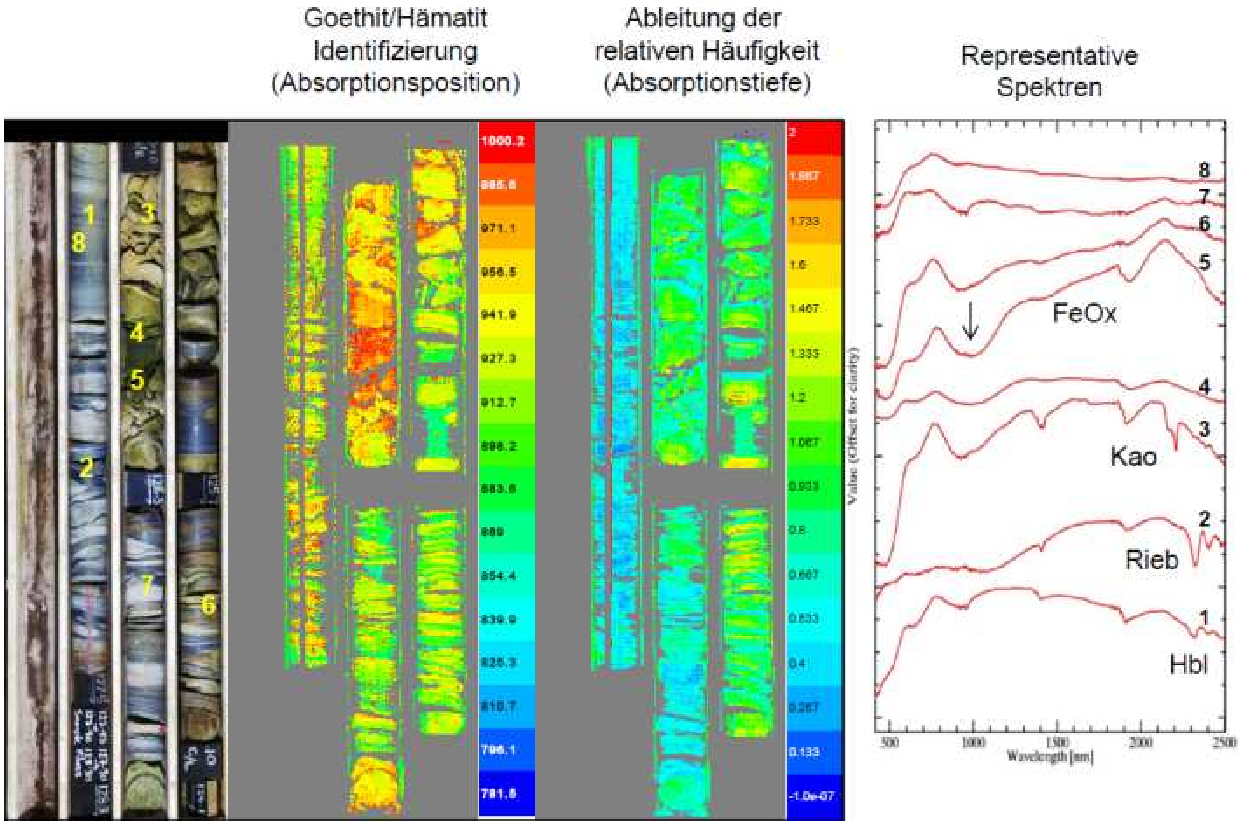
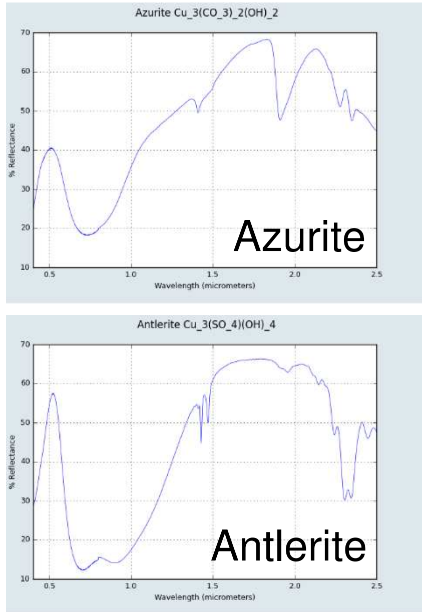
Source: M. Schodlok (BGR)

Hyperspectral imaging (2/2)





Aster spectral library
(Baldridge et al 2009)

Source: M. Schodlok (BGR)

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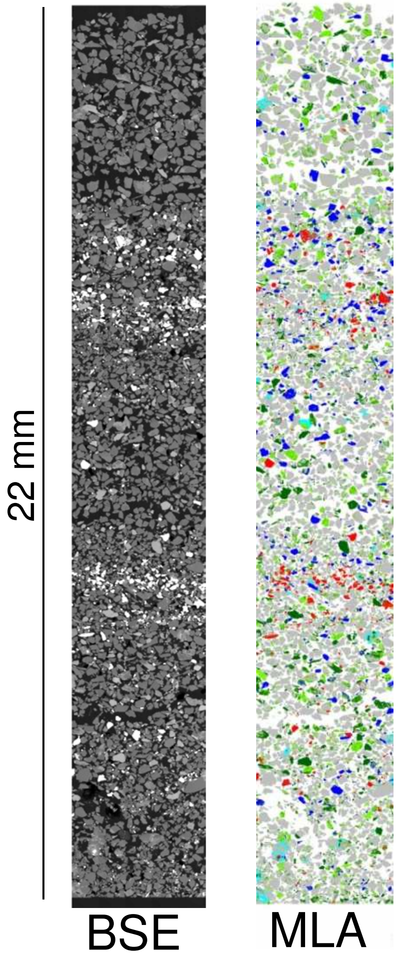
Case study: weathering of As-rich mining residues (1/3)

poly-metallic sulfide mine district, Freiberg (Germany)

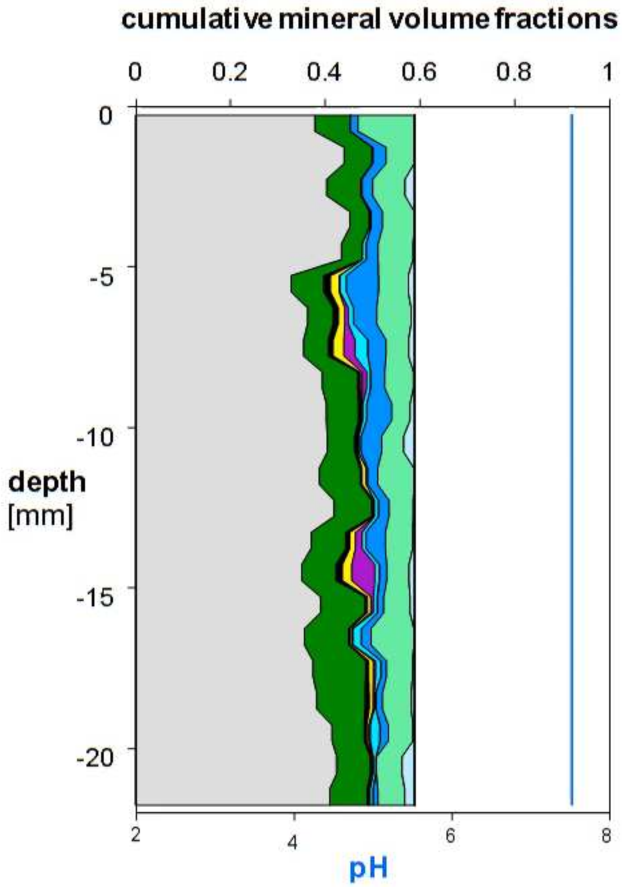


Quantitative mineralogy:
Scanning Electron Microscopy (SEM)
Mineral Liberation Analysis (MLA)

- sulfides (e.g. pyrite, arsenopyrite) (1.84%)
- mica group, chlorites (6.10%)
- carbonates (3.45%)
- non-reactive (37.9%)
- pores (44.0%)
- Gels (e.g. Fe-(As)rich), clay minerals (5.81%)
- gypsum, sec. sulfates (0.86%)

Source: M. Redwan (BGR)

Case study: weathering of As-rich mining residues (2/3)



Reactive-Transport simulation of cemented layer formation

Primary phases:

- ankerite
- siderite
- arsenopyrite
- pyrite
- arsenian pyrite
- biotite
- non-reactive phases
- pore space

Secondary phases:

- alunite
- gibbsite
- iron arsenate (am)
- ferrihydrite
- jarosite
- calcite
- gypsum
- silica gel
- kaolinite

Time (years)

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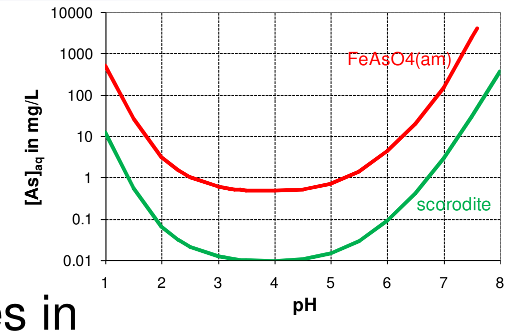
Case study: weathering of As-rich mining residues (3/3)

What happened?



Oxidation of sulfides
(release of H^+, Fe, As, SO_4)

↓

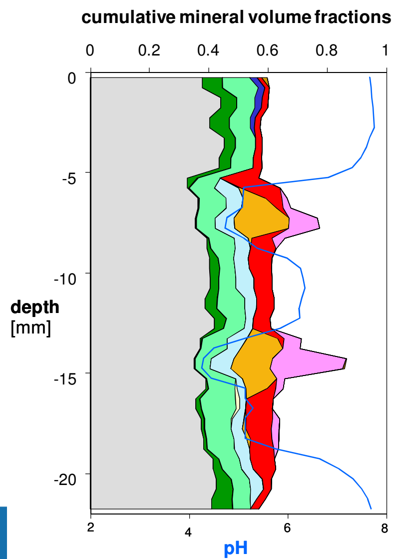
Dissolution of carbonates
(release of Ca, Fe, Mg, HCO_3)

↓

Precipitation of gypsum & Fe-gel

↓

Metal sorption



→ Depletion of carbonates in sulfide-enriched layers

↓

Significant pH reduction

↓

Increased biotite dissolution
(release of K, Fe, Mg, Al, Si)

↓

Precipitation of jarosite and FeAs-gel (high V_m!)

↓

Formation of cemented layers

Conclusions

- Scanning methods provide chemical, mineralogical, textural and geological information on a multiscale level.
- Methods are fast and non destructive.
- Methods need almost no sample preparation and operate at low cost.
- Evaluation can be automated for various rock and ore types.
- They offer innovation possibilities, e.g. in prospection, exploration, processing
- Applications in the field of Cu-As-ores/residues need to be developed.

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