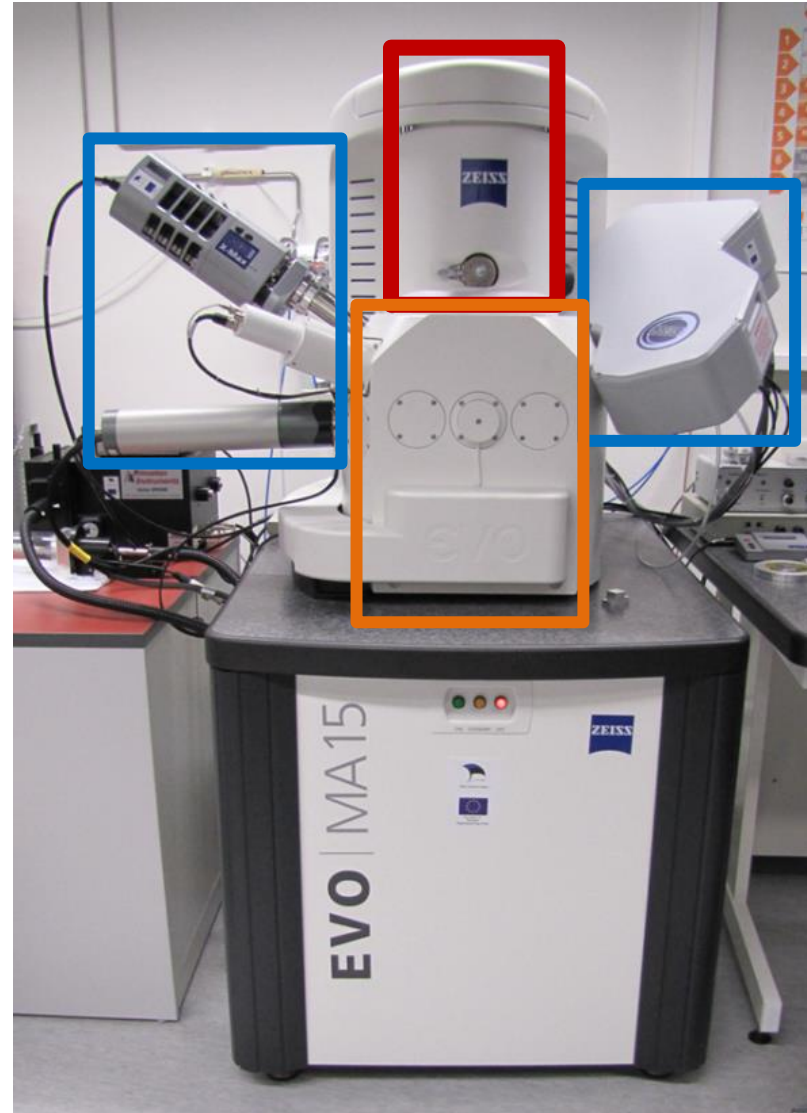


General aspects of Scanning Electron Microscope Energy- dispersive X-ray Spectroscopy (SEM-EDS)

**Päärn Paiste, PhD
Research Fellow in Geology**

Principle components of SEM-EDS

- Electron gun and electron optics – generates and guides the electron beam
- Sample chamber – samples under vacuum conditions
- Detectors – for imaging and chemical composition analysis



How an SEM-EDS works

Sample surface is bombarded with high energy (tens of keV) **Primary electron (PE)** beam in a scanning pattern

The primary electrons interact with the sample surface and produce **Secondary (SE)** and **Backscattered electrons (BSE)**

SE and **BSE** are detected and are used to generate an image of the sample surface

The high energy **Primary electrons** are also energetic enough to excite the elements present in the sample, causing them to emit **X-ray photons characteristic of a specific element**, which can be detected and used to determine the elemental composition of the sample

For a more detailed overview of X-ray emission: [Introduction to XRD and XRF methods](#)

What samples can be analysed with SEM-EDS?

Essentially **all solid samples**, that can fit into the sample chamber can be analysed – from a grain of sand to a bronze axe

The samples can be viewed at high magnifications (up to 100 000x) – **we can see μm sized features**

However:

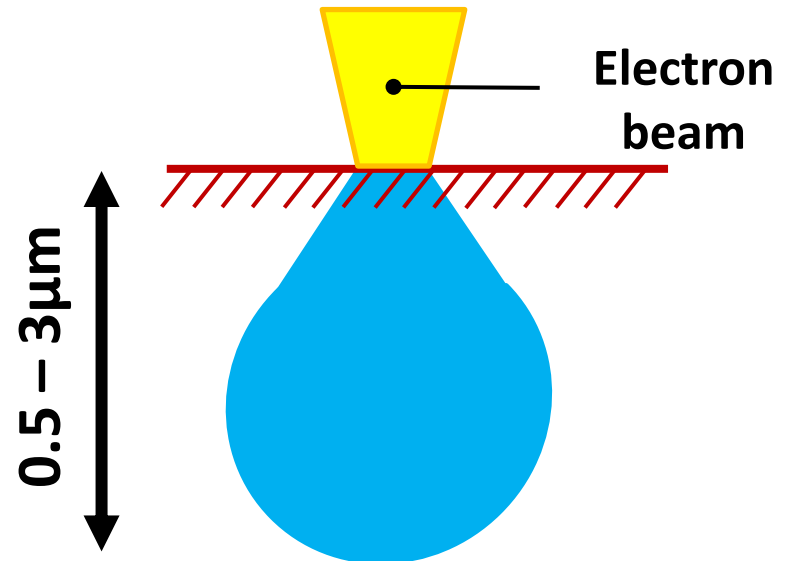
- The sample **must not contain any volatile compounds** – the samples are under vacuum conditions during analysis
- The sample **should be conductive** – **if not**, the samples must usually be **coated with a thin layer of Carbon, Gold or Platinum**
- The analysis is **non destructive**, but too high electron beam energy **can etch a small hole** in some samples -

For a more detailed overview : [Analysis with SEM-EDS method](#)

Limitations of SEM-EDS

- The detection limits of SEM-EDS are around **0.1%** and we **can not measure elements lighter than C** (B, Be, Li, H)
- There are numerous **spectral overlaps for different elements** – for example S peaks overlap with Pb and Hg
- Sample **composition** (matrix) **influences the analytical signal** – same Pb concentration gives different signal in paper vs bronze

- The elemental composition signal **does not originate from the sample surface but from within - around a few μm depth**



Limitations of SEM-EDS

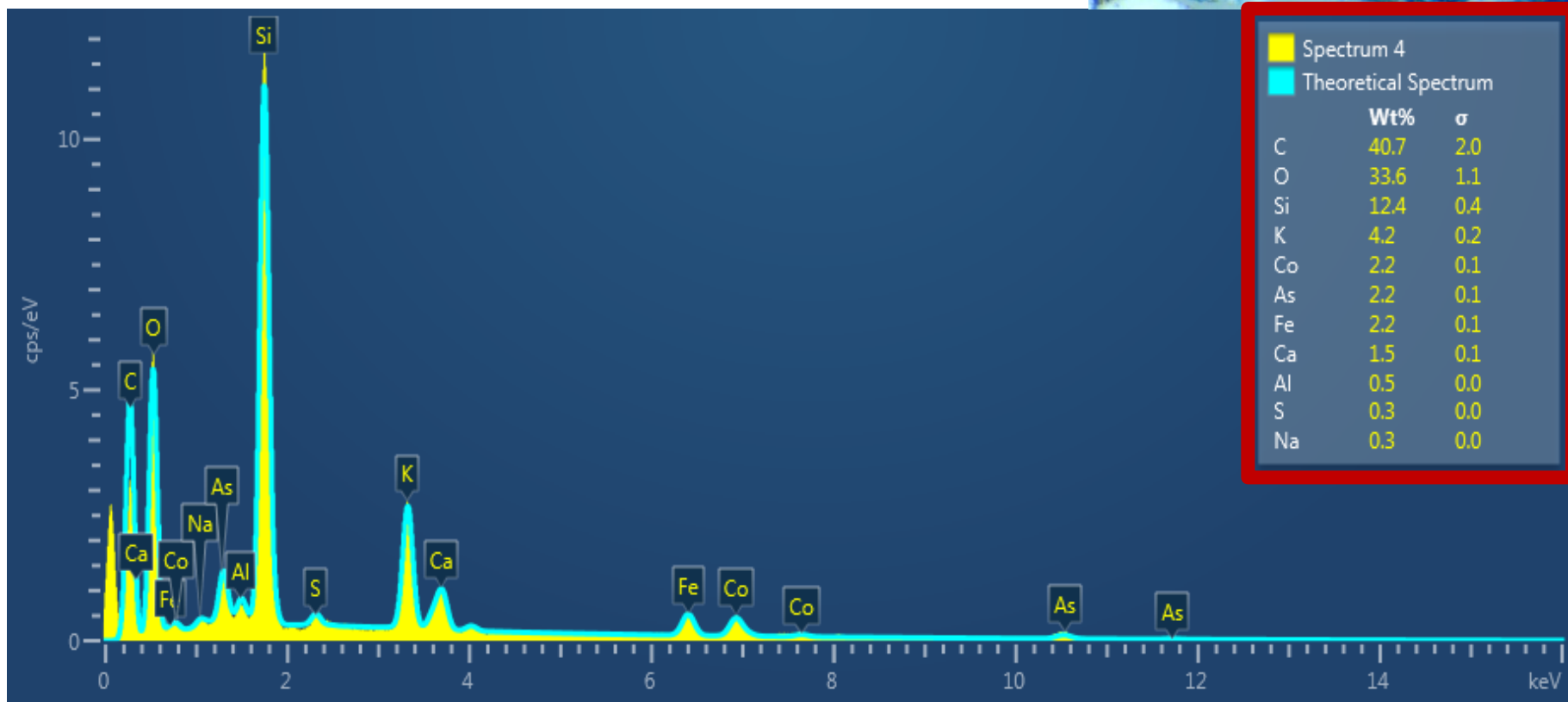
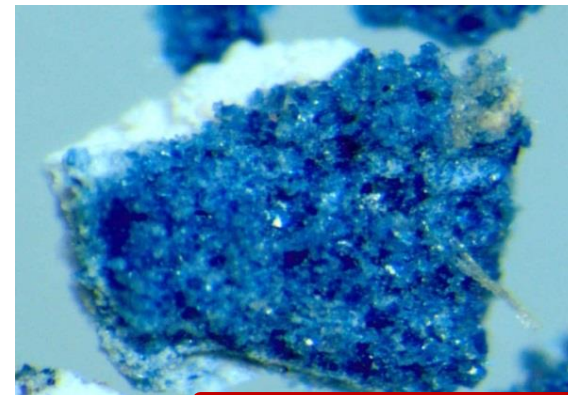
- EDS analysis is a semiquantitative analysis - provides an estimation of the chemical composition!
- Quantitative elemental composition data can be obtained if SEM is equipped with a Wavelength Dispersive detector - WDS (SEM-WDS, Microprobe or Electron Microprobe)
- To gain quantitative results or improve accuracy of semiquantitative analysis matrix matched (with similar composition to samples) calibration standard must be used

Examples of SEM-EDS analysis – single spot

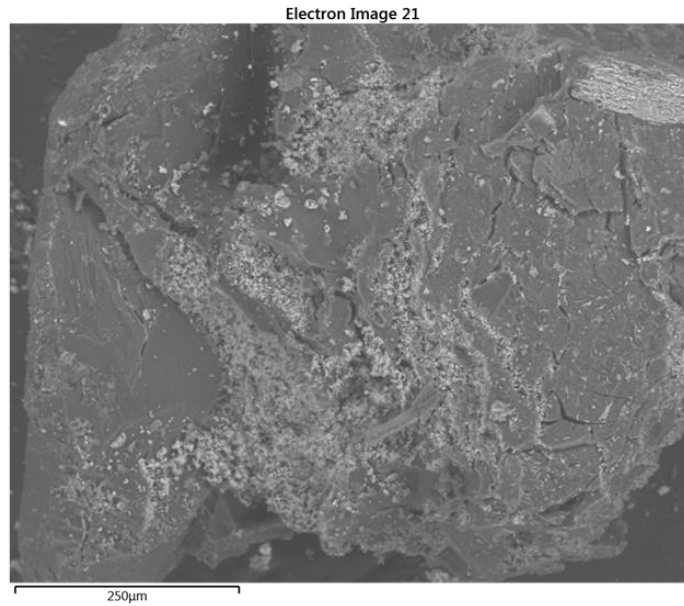
Identification of blue pigment in a wall mural



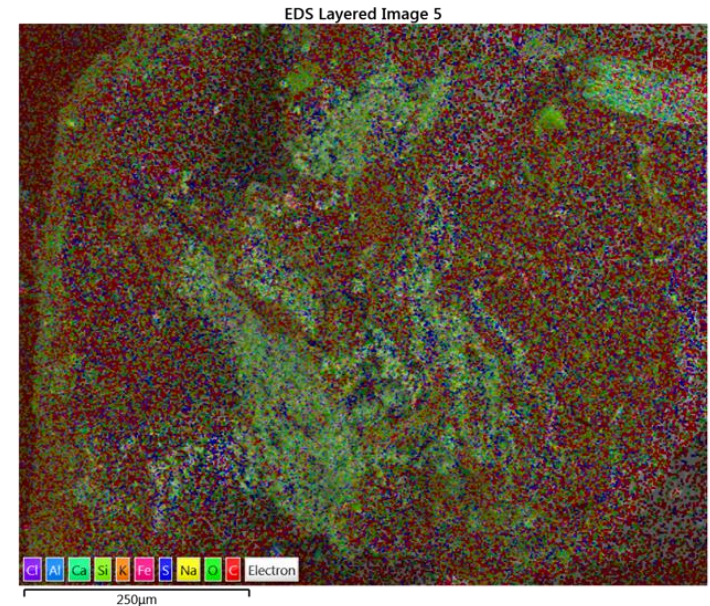
Smalt ($\text{SiO}_2 \cdot \text{K}_2\text{O} \cdot \text{CoO}$)



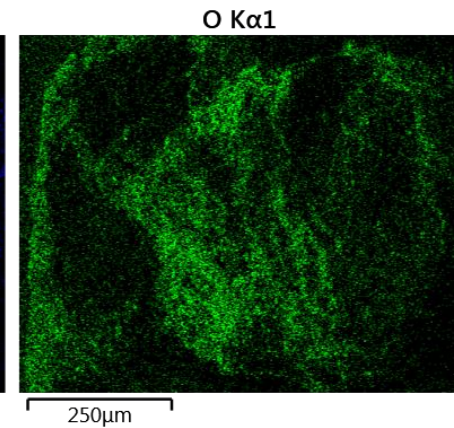
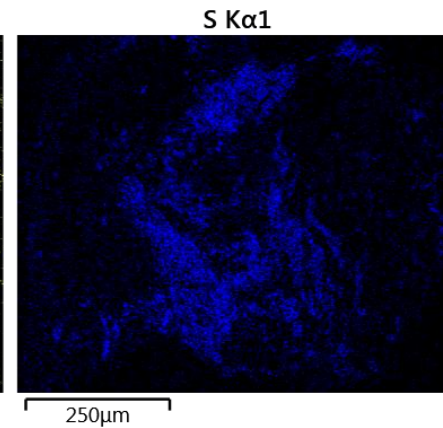
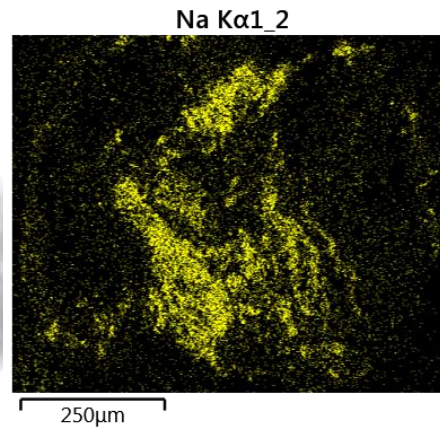
Examples of SEM-EDS analysis – elemental mapping



SEM image



Combined EDS elemental distribution map



Single element distribution maps

Summary

Very small samples can be analysed fast with minimal sample preparation

Non destructive method of analysis

Can be used to measure the elemental composition of **small areas** or create an **elemental distribution map**

Provides approximate concentration and distribution patterns of **Major constituents** in the sample

SEM-EDS is a **semiquantitative** analysis but SEM-WDS (or Microprobe) is **quantitative**