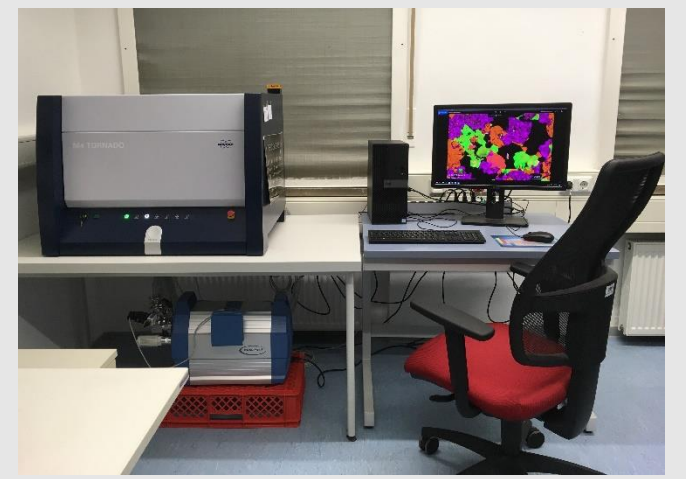


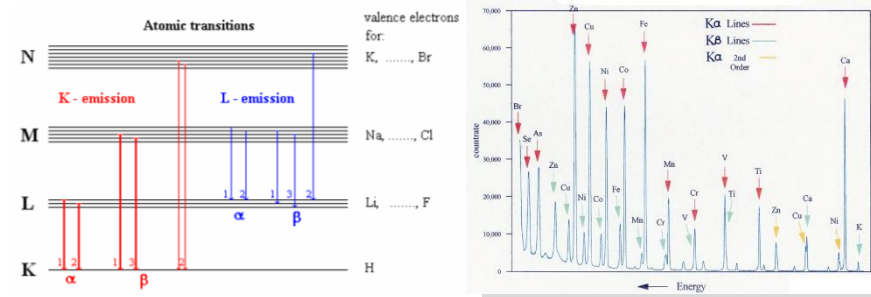
The μ -XRF instrument: Principles, Applications and Case Studies

Christoph Heubeck

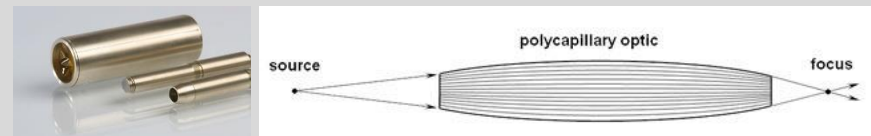


Concept

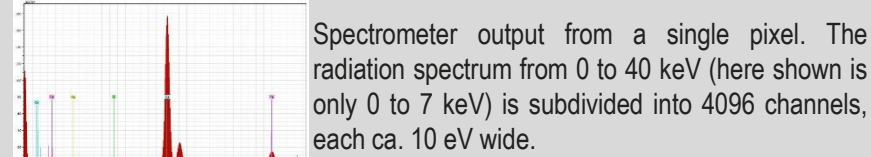
We have been using since 2018 a Bruker Tornado M4 micro-X-Ray Fluorescence Instrument (the " μ -XRF"); it is administered by the working group Allgemeine und Historische Geologie (Prof. Heubeck). This is a benchtop instrument consisting of the X-ray chamber (about the size of a large microwave oven), a desktop computer with screen, and a vacuum pump. The instrument is a scanning device, that is, it produces maps composed of individual pixels, each of which contains the information gained from fluorescence spectra which elements emit when irradiated by X-rays. The x-ray head is arranged vertically and fixed; the object moves underneath the head on a motorized automatic stage.



When electrons in the orbit around nuclei are excited by radiation, they are briefly "lifted" to higher orbits (K-, L-, M-, N-bands). When they fall back to their original orbit, they emit a characteristic radiation which is specific to the energy difference between the orbits. The K α -line is the most commonly measured peak, named for the fall-back of an electron from the α orbit of the L-band to the K-orbit.



Focusing the x-rays on small areas (the minimum pixel size of the M4 Tornado is 20 μ m) requires so-called capillary optics: x-rays are guided within a bundle of carefully arranged micron-scale glass tubes. This was a breakthrough technology which made it possible to finely focus X-rays to impinge on the sample surface while maintaining acceptable beam brilliance.



Spectrometer output from a single pixel. The radiation spectrum from 0 to 40 keV (here shown is only 0 to 7 keV) is subdivided into 4096 channels, each ca. 10 eV wide.

Each pixel generates an spectrum of electromagnetic frequencies at various wavelengths. For each pixel, the instrument collects the entire reflected fluorescence spectrum, deconvolves it and assigns various elements registered in the computer's element spectra library to the spectra. The element abundances in each pixel can be quantified but also simply be shown in variable color intensities.

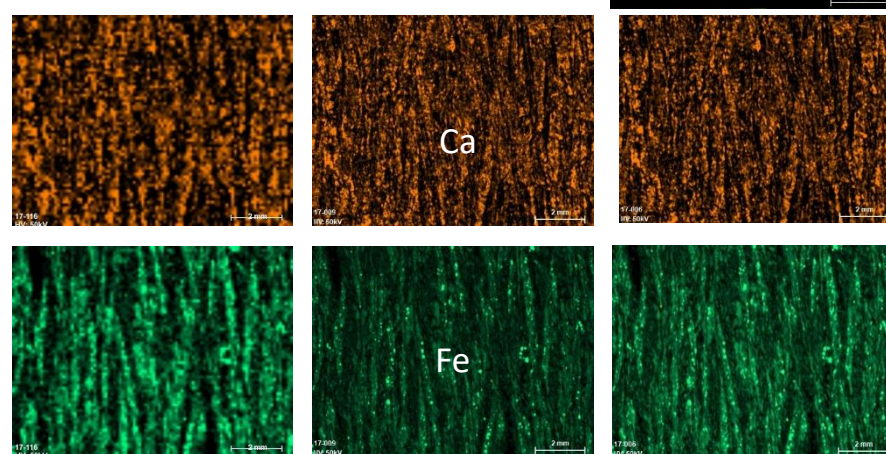
The instrument operates in point, line or area mode. Typical measurement time per pixel is 25 μ sec. A map of 1000 by 1000 pixels, done twice, requires about an hour of measurement time.



Samples should be flat, level, clean, and thicker than the penetration depth of the x-rays (element-dependent; about 20-100 μ). Liquids and moist substances (leaves, roots, food) can be measured, too, but we would not run the vacuum pump, so there would be some interference with and absorption from gases. Sodium (Na) is the lightest measurable element (no, C, O, N, sorry !)

Comparison of low- and high-res acquisition with short- and long-term exposure

A pale grey-green chlorite-actinolite-schist (KVT-BGB contact; sample CH 17-006); 11.3 ~ 8.43 mm



5:04 min; 100*75 px pixel; 7500 px; size 113 μ ; 10 μ sec/pixel
 55 min; 500*373 px; 186500 px; size 23 μ ; 10 ms/px
 5:44 hrs; 500*373 px; 186500 px; size 23 μ ; 40ms/px

The instrument is very useful wherever the display of spatial element (not mineral !) distribution on a mesoscale is needed. The resolution of 20 μ means that medium-sand grains, ooids, thick cements, coatings or microfossils (ca. 300 μ diameter) can be shown by about 10 to 20 pixels in each direction. The arrangement of such features, such as lamination, fine-scale bedding, heavy mineral lags, veining, pygmatic folding, gneissic schistosity etc. can also be visualized clearly.

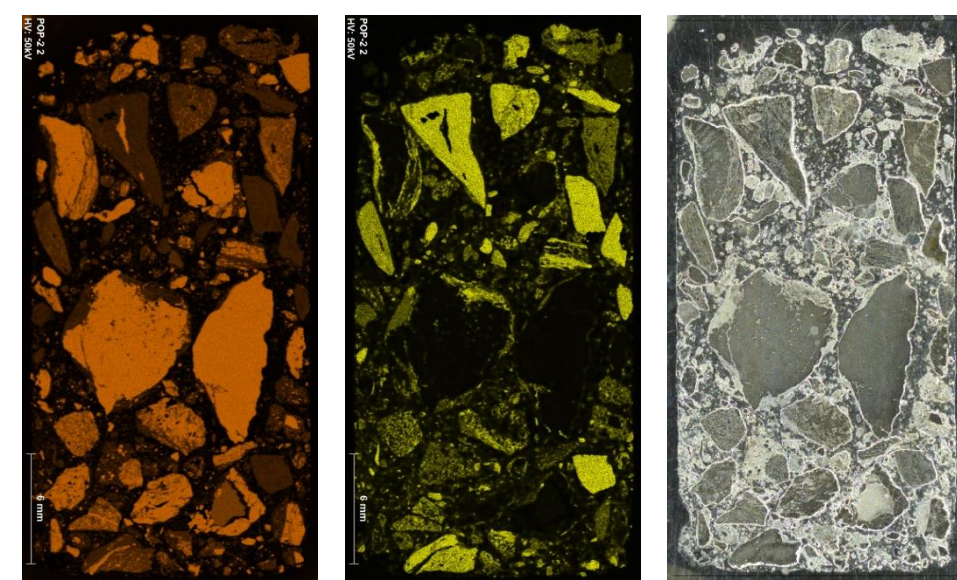
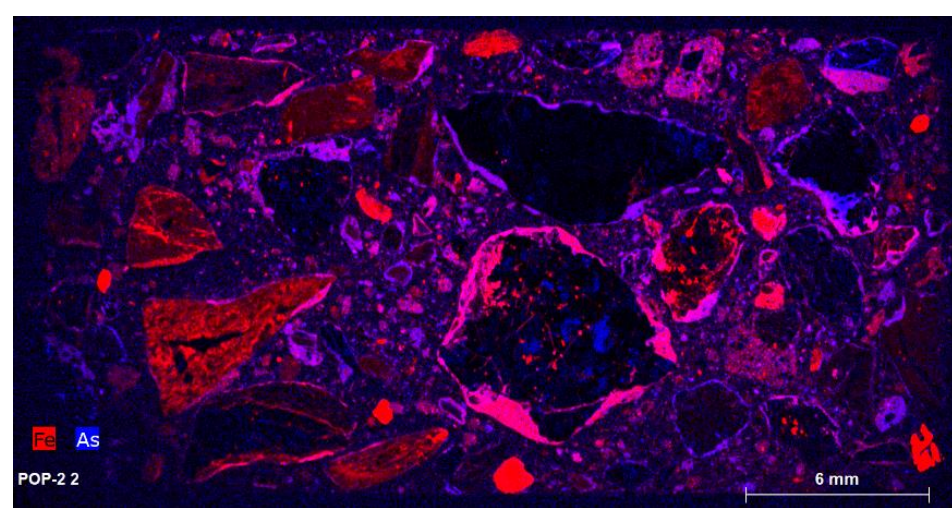
Features



Fish-eye view of the sample chamber. A thin block of a rock sample lies on the clear polyacrylic stage at camera level. The black conical head houses the x-ray lens. The focal distance is a few mm.

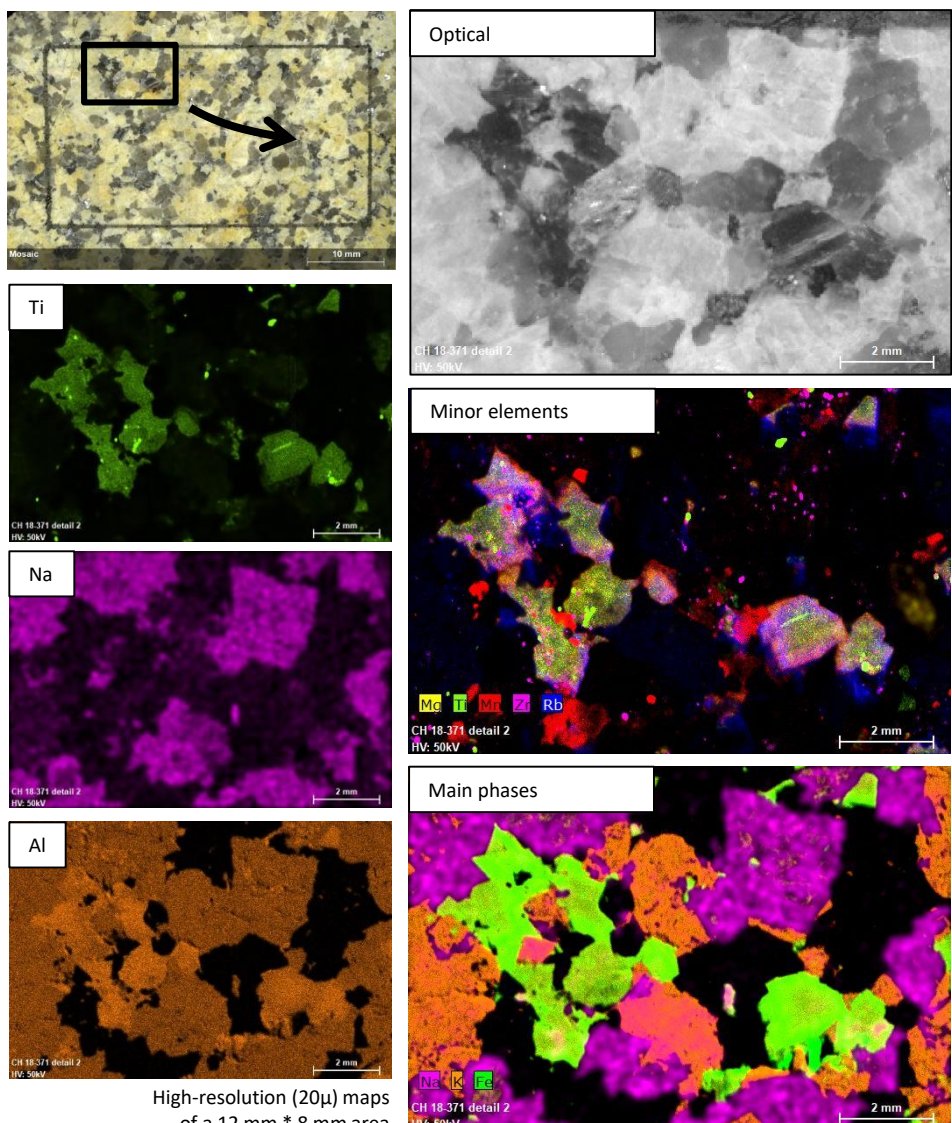
The maximum possible size of a sample is about paperback size. Most often, however, we use the block samples required for thin section preparation (ca. 36*24 mm) and select a representative region.

Thin sections are too thin for analysis because the penetration depth of the x-rays is high enough to measure the composition of the carrier glass (Na, Si, B, K etc.) and, if present, the cover glass.



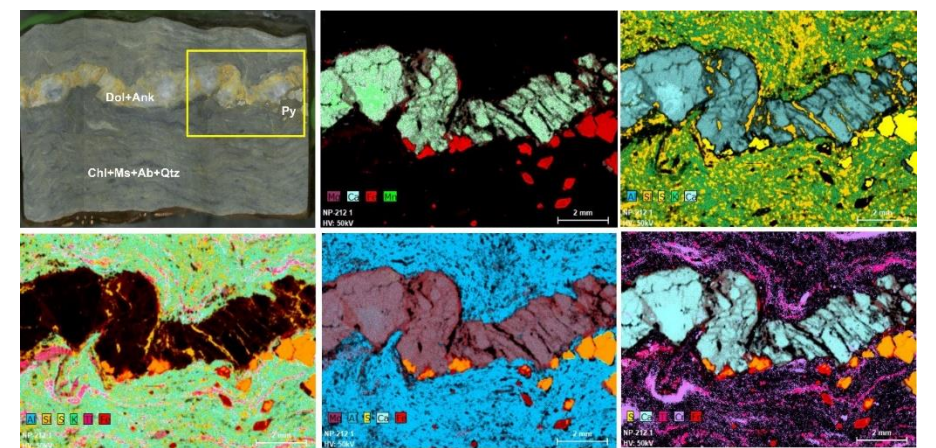
An arsenic-bearing hydrothermal carbonate breccia from Prof. Juraj Majzlan's analyses.

The Henneberg Granite (yellow variety): Fischer Granite quarry, Thüringer Schiefergebirge

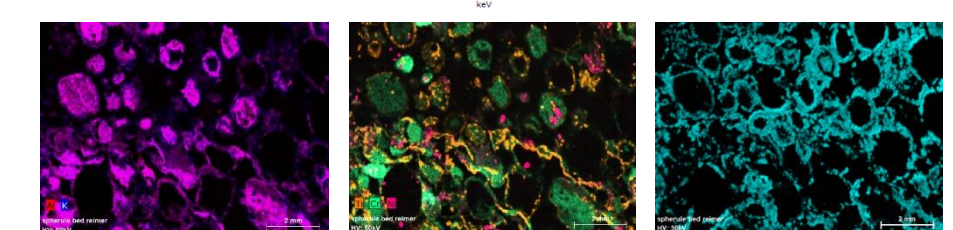
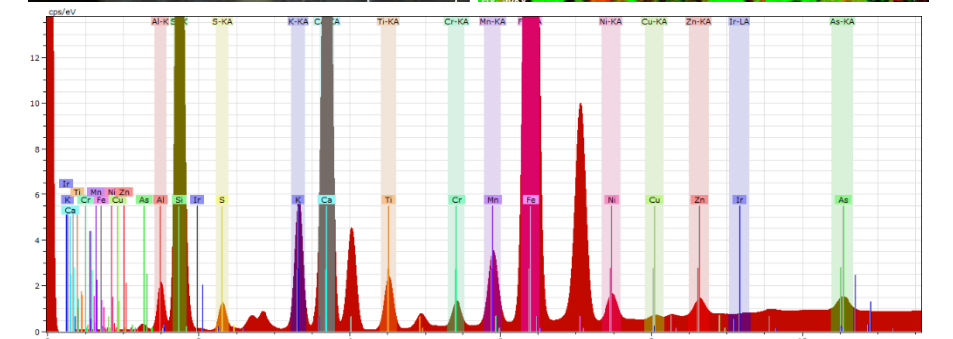
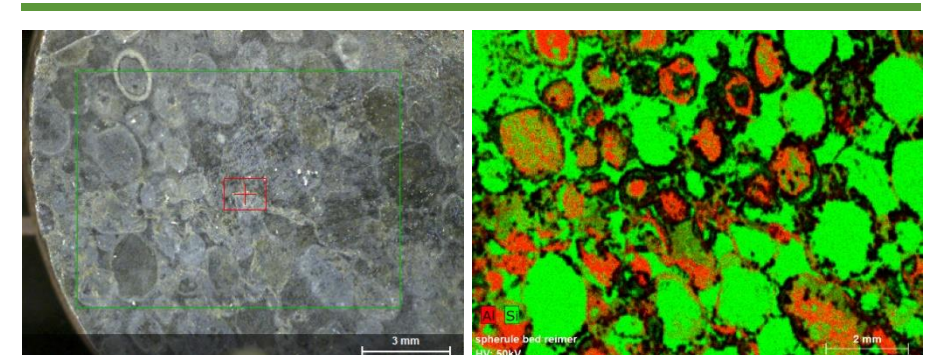


High-resolution (20 μ) maps of a 12 mm * 8 mm area

Case Studies



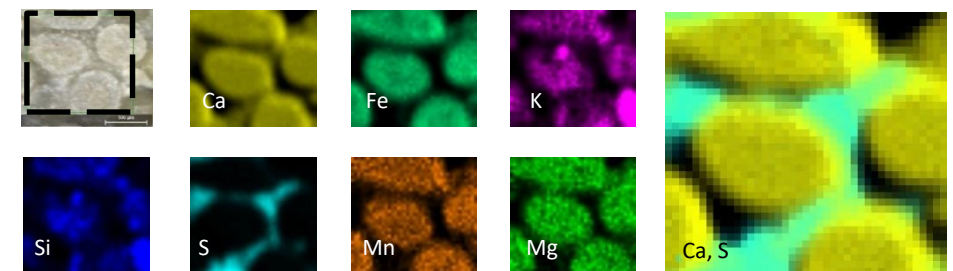
Elemental maps of crenulated phyllite sample NP-212 (Msc, Phumelele Mashele, Univ. Witwatersrand 2021) showing euhedral pyrite (Fe, S) and titanite crystals (Fe, Ti) associated with hinges and kinks of a dolomitic (Ca, Mg) vein within a fine-grained quartzofeldspathic (Al, Si, K) and mafic (Cr, Fe) siliceous matrix.



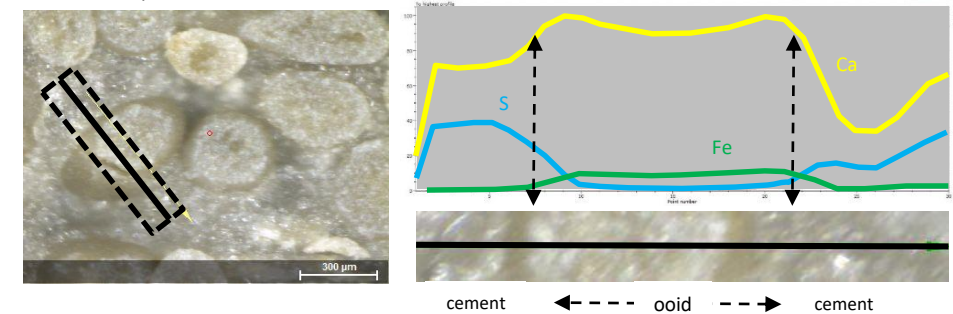
A polished core plug of spherule bed S2 or S3 from the Archean Barberton Greenstone Belt, South Africa. This is a sandstone composed of orbitally transported melt droplets which were generated by a distant, very large meteorite impact and rained down into deep water. μ -XRF reveals the ultramafic target material containing high Cr and Ni but NAA of some samples show extremely high Ir values.

A gypsum-cemented oolitic grainstone (sabkha-facies of Grundgips; middle Keuper (upper Triassic); Burgberheim/Central Franconia; CEH 2018)

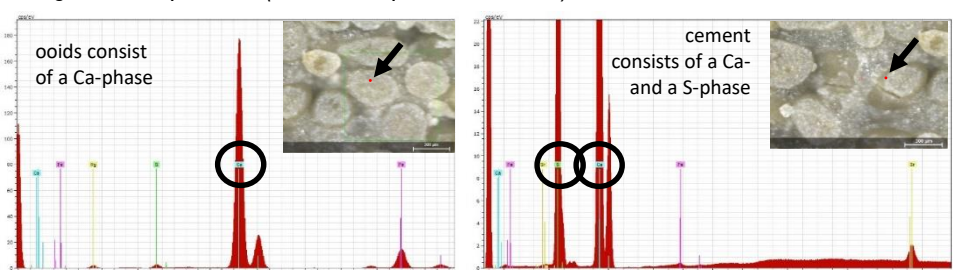
Elemental maps (ca. 60 * 60 pixel; ca. 1 mm * 1mm)



Line analysis across a carbonate ooid and its surrounding gypsum cement (30 points along 600 μ)



Single-Point Spectrum (red dot at tip of black arrow)



The actual spot size depends on various design parameters of the polycapillary lens and on how close the lens can be positioned relative to the sample surface; therefore, the flatness of the sample plays a role. In principle, it is possible to investigate materials of several mm relief but we usually use level and flat surfaces.