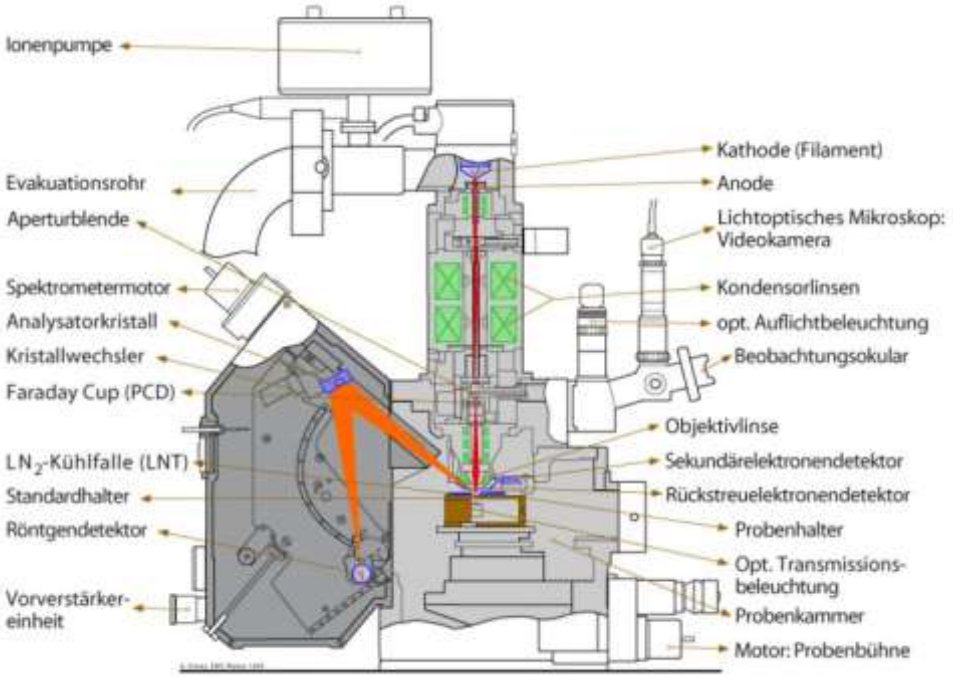
Electron microprobe

The electron beam microprobe is referred to as EMP (electron microprobe) or EPMA (electron probe microanalyser). It is essentially a scanning electron microscope to which spectrometers have been attached for the quantitative analysis of concentrations of main, secondary and trace elements. This allows the composition of even the smallest areas to be analysed without destruction, i.e. even mineral grains measuring just a few micrometres can be analysed in situ (i.e. in the rock matrix) with high accuracy. In addition to rock samples, even the smallest glass particles, gemstones, bones, teeth, skeletons of marine organisms and much more can be examined.

The basic principle

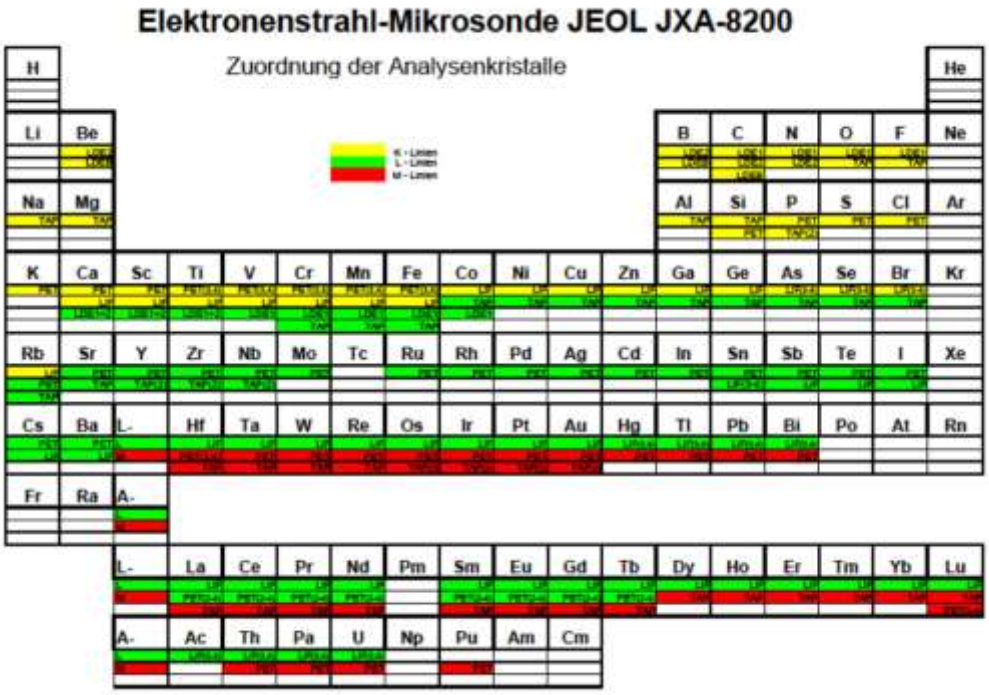
Electrons are thermally generated at a tungsten cathode and accelerated towards the sample in a high vacuum by an electrical potential of up to 30 kV. On their way there, the charge carriers pass through a series of electromagnetic coils that shape and focus the beam before it hits the sample. There, they interact with the atoms of the different elements in the sample, which are excited to emit their specific X-rays. Depending on the acceleration voltage, the interaction volume is only a few cubic micrometres. X-ray spectrometers function like X-ray filters and measure the number of X-ray pulses for a specific element sequentially (normalised to the measurement time and the beam current). The measured intensities are then compared with measurements on reference materials (whose compositions have been well determined beforehand) and used to calculate element concentrations. The matrix correction performed after the measurement is necessary to correct for the mutual influence of the elements on each other.



Schematic structure of a microprobe: The electron beam is shown in red, the proportion of X-rays hitting the counter tube is shown in orange.

Which samples can be analysed?

In principle, the EMS can be used to analyse the elements from Be to Pu (see periodic table) in all solid materials, provided that they are vacuum-stable and do not change under the influence of the electron bombardment. For quantitative analyses, a very good surface polish is also mandatory. Non-conductive materials must be coated with a 15-20 nm thick carbon layer to prevent negative charging of the sample.



Equipment in detail

Imaging

In addition to a secondary electron detector, a backscattered electron detector and a cathodoluminescence detector are available. Furthermore, thin sections can be examined under a microscope with an optical camera in reflected and transmitted light.

Analytics

Two of the five wavelength-dispersive spectrometers work with Ar/methane flow meters, which are optimised for measuring light elements in combination with LDE1, LDEB, LDEC, TAP and TAP and PET crystals. The other spectrometers are equipped with Xe counters and a combination of PET and LIF crystals. The EDS system makes it possible to measure several elements simultaneously and thus perform phase identification in a matter of seconds.

Preparation of an electron beam microprobe analysis

Sample preparation, documentation and plan for quantitative analysis

1. **Casting and polishing of solid samples**
    1. Samples are mounted/cast in suitable holders to meet the requirements of the microprobe stage. This can be carried out by commercial thin section laboratories.
    2. Samples are polished step by step to a final polish of 0.25 µm and cleaned of any remaining polishing material and oil.
    3. Excess epoxy resin is removed so that the samples fit into the referenced microprobe holder.
    4. The samples are cleaned if necessary.

2. **Sample photography and documentation**
    1. If a detailed EPMA is to be performed, an overview image of the sample is required. This can be a digital camera image or scan with relatively low magnification; for analyses with high magnification, a photo mosaic is required.
    2. With EPMA, samples are examined using reflected light, secondary electron and backscattered electron images. Transmitted light is not usually used, meaning that transmitted light images are of lesser value than reflected light images. Reflected light or REM-BSE photo mosaics, which can be prepared and organised in a graphics program, are therefore ideal.

3. **Carbon coating of samples**
    1. The samples must be coated with carbon before microprobe analysis. Please do this before the day of the session.
    2. If the samples are not clean, contain oil residues or have fingerprints, they must be cleaned before coating. All coating work is carried out with gloves.
    3. The target thickness of the carbon is ~ 22 nm. This corresponds to the red-blue colour of the polished brass reference material.
    4. Follow the specific instructions for carbon coating in the laboratory.
    5. Coated samples should be stored in a desiccator as required.

4. **Priority plan for the EPMA session**
    1. The electron microprobe is typically used for SE and BSE imaging, element mapping and quantitative microanalysis. You must therefore determine in

advance the focus of your research, the priority of the tasks and the time required on the instrument.

2. Efficient use of the microprobe may require flexibility in your schedule. Once the instrument has been calibrated, you should be prepared to work longer hours to complete your work. Please do not assume that your work can be extended to the following days.
3. Quantitative analyses require a considerable amount of time (usually 2 hours or more) and should be performed in a single run whenever possible. These runs include manually recorded points and transition to automatic analysis during night runs.
4. Element mappings are usually performed overnight and also require a significant amount of setup time.

5. Quantitative electron beam microprobe analysis

1. Microprobe analysis requires relatively short X-ray counting times for major elements (approx. 10-100 wt.%), longer times for minor elements (approx. 1-10 wt.%) and significantly longer times for trace elements (<1 wt.%).
2. A list of the expected element inventory should be prepared before the microprobe analysis and discussed with the operator.