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THE ADVANTAGES OF EDS OVER WDS FOR BEAM-SENSITIVE MINERAL ANALYSIS

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Electron microprobe (EMPA) equipped with wavelength-dispersive x-ray spectrometers (WDS) is the most commonly used method for the quantitative non-destructive analysis of minerals at µ or sub-µ scale. Can energy-dispersive spectrometry (EDS) compete with WDS in quantitative analysis?
While trying to analyze experimentally grown gagarinite we found WDS limitations caused by extreme volatile loss (50 % of Na loss in ~7.5nA·s, with an artificial increase in subsequently analyzed elements) from e-beam interaction with the material. We turned to full-sized standard-based EDS on SEM as an alternative, which allowed us to overcome the volatile loss problem. For successful EDS analysis the main forced-restrictions of EPMA need to be strictly followed: use clean and pure standard references coated with the same element and thickness as the unknown; use the same geometry for spot analysis of standard and unknown (identical "working distance", the analysis only with centrally parked/non-scanning electron beam), the equipment should have the ability to measure the beam current.
The free (public domain) NIST DTSA-II software was used for EDS quantitative recalculations into chemical concentrations. To overcome the volatile loss, 30 EDSes with a dwell time of two seconds and a beam current of 0.605 nA for each were acquired from previously not analyzed gagarinite grains. EDS spectra were summed, forming a single representative 60s EDS, which was further processed with quantification.
EDS-based quantitative results for gagarinite have no Na loss and no increased concentrations of other elements due to Na loss. To insure method correctness as compared with the well-established WDS mineral quantification results, the EDS of albite (interference-free), narsarsukite (few mild interferences), chevkinite, and britholite (strong multiple spectral interferences) were acquired, quantified, and compared. For major and minor elements the results from EDS are on a par with the WDS results (albeit WDS is still the "king" considering the trace quantification or high beam currents). The main advantage of EDS is the same (or better, for beam sensitive samples) precision and accuracy for major elements with lower beam current and shorter analytical time. EDS is generally more available - commonly scanning electron microscopes are equipped with full-size (or large size) single or multiple EDS detectors, with signal processing units (SPU) equipped with the highest spectral resolution options available for low count rates and low beam-currents. For comparison, the EPMA commonly are equipped with only the single and downsized EDS (due to space constraints), where SPU is tailored for much higher beam currents. For beam-sensitive minerals, the SEM-EDS equipment has a clear advantage over EPMA-WDS. It should be mentioned that there are possibilities to combine both (i.e. major and minor element k-ratios measured on SEM-EDS with k-ratios of trace elements on EPMA-WDS), albeit currently, available combination techniques are very cumbersome.

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