

Improvements for quantitative WDS and EDS analysis of minerals: Validation of standard reference materials and technology-led optimisation of analytical parameters

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Wavelength Dispersive Spectrometry (WDS/WDX) and Energy Dispersive Spectrometry (EDS/EDX) are analytical techniques that require standard materials of known composition to be measured to produce accurate quantitative compositional data for unknown samples. However, a problem exists that many of the published compositions for standards have often been determined by EDS/WDS in the first instance and have not been determined/verified by other methods. To try and address this issue, this study presents an approach for establishing and validating the composition of mineral standards.

The methodology involves choosing simple primary standards (e.g., pure elements, stoichiometric binary compounds), and analytical conditions (e.g., accelerating voltage (kV)), with the most minimal matrix corrections for the standard being verified. Figure 1 shows the matrix correction between different pure element/compounds and mineral standards at different kVs. Based on such evaluations, optimal primary standards and kVs can be chosen for determining the composition of complex mineral standards. This approach is evaluated by analysing orthoclase and forsterite standards using a Wave WD spectrometer and Ultim Max EDS detector on a FEG-SEM at different kVs and comparing the measured to published elemental concentrations. Cation totals were also calculated to check they were consistent with the mineral formula, both for the total and each cation site.

To conduct the standard analysis a technology-led approach was used to determine the analytical parameters required to achieve a certain precision on each element being measured by WDS. The optimization engine calculates a theoretical WDS spectrum based on the standard composition and the SEM beam current. From this WDS backgrounds are automatically positioned to avoid peak overlaps, and peak and background counting times are calculated to achieve a certain level of precision. This technology-based approach removes the requirement for trial-and-error analyses and speeds up the measurement of both standards and unknowns for users with all experience levels.

Figure 1. Matrix corrections between different pure element/binary compounds and mineral standards, for Si at 15 kV (left) and Fe at 20 kV (right). A value of 1 on the line tying standard (start) and unknown (arrow end) represents no matrix correction. Matrix correction values below 0.8 are omitted.

