

## THE DETERMINATION OF THE IRON OXIDATION RATIO IN MINERALS WITH THE ELECTRON MICROPROBE- IS IT WORTH THE EFFORT?

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A drawback of mineral analysis with WD-EPMA is that the oxidation state of Fe is not obtained. Oxidation states can be estimated using the number of oxygens in the ideal mineral formula as a normalization constraint. However, this method fails where mixed valence states occur, where specific sites may be only partially filled, or where additional anions (OH, F, Cl, O<sup>2-</sup>) occur. In these cases oxidation states can only be estimated within broad ranges. Fe-L-line X-ray spectroscopy in TEM and EPMA has found renewed interest as a method for determining the Fe<sup>2+</sup>/Fe<sup>3+</sup> ratio with high spatial resolution. The flank method (Hoefer et al. 1994) for EPMA first defines X-ray emission spectra of Fe<sup>2+</sup>- and Fe<sup>3+</sup>-rich samples and critical energy ranges of maximum intensity difference. These critical regions are used to calibrate the system on the basis of samples with known Fe<sup>2+</sup>/Fe<sup>3+</sup>. We have successfully applied the flank method to amphiboles. The information obtained can be used with high accuracy (i) to classify amphiboles on a micrometer-scale, (ii) to resolve the oxidation state in zoning patterns within crystals, and (iii) to optimize stoichiometry calculations for amphiboles on a variable anion basis, thus allowing relatively precise estimates of the (OH)-content. The flank method requires very accurate and time-consuming calibration of the microprobe used and a reliable set of standards. It is not a routine method, but it is an invaluable tool for specific applications.