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Appearance, study and a possible correction for boron: Insights on ultra-soft X-ray measurements using a synthetic multilayer crystal and the EPMA

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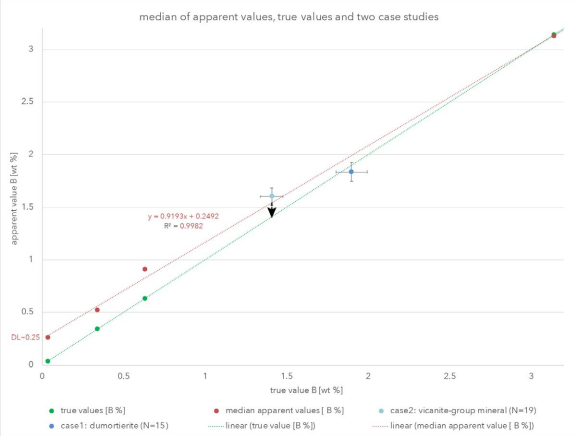

Boron (B) is a light lithophile element. Together with lithium, boron (and its isotopes) gained attention for its role in hydrothermal fluids to track the circulation of matter down to the deep mantle. In quite a lot of nominally boron-free phase, boron is detected in small quantities of some hundred or thousand ppm.

Boron can be chemically separated from its silicate matrix in minerals and rocks by alkaline fusion and the concentration determined thereafter from the solution by e.g. ICP-MS. In-situ, very small concentrations of boron can be analysed by SIMS. Another extremely effective and inexpensive, albeit less precise way to analyse boron is with the electron microprobe (EPMA).

When measuring boron in tourmalines, or in minerals of similar boron concentration, calibrated with the Harvard schorl, no deviations in the peak intensities could be detected with a proven analysis protocol and using the Mo/B4C multilayer crystal LDEB. Only when boron is detected in natural and experimental samples with significantly lower boron concentrations than in tourmalines, irregularities in the analysis become visible. This phenomenon has been analytically investigated.

A voltage of 10kV and a current of 20nA using 10µm probe size was applied for EPMA boron analyses. The acquisition time of the peak was 60s, and 30s each for both background level. Backgrounds can be set best by scanning the unknown phase at the boron peak position.

Using four natural and artificial solids with boron concentrations from 0.035 to 3.14 wt.% B, an apparent linear trendline was drawn.

The intersect of that trendline with the y-axis represents the detection limit of boron at about 0.25 wt.% B. The discrepancy between the apparent and the true value trendline at boron concentrations of 0.25-2.1 wt.% B, shows that a correction is necessary [1]. At higher boron concentrations, the discrepancy between the apparent and true value trendlines is within uncertainty of EPMA analyses and disappears completely at about 3 wt.%.

[1] Wilke, F. D. H. (2023). Appearance, study and a possible correction for boron: a phenomenon in ultra-soft X-ray measurements using a synthetic multilayer crystal and the EPMA. *European Journal of Mineralogy*, *35*(1), 59-64.