Optimisation of light element measurement with high resolution wavelength dispersive spectrometry

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Since the development of multilayer dispersive elements with large periods, the electron microprobe is a powerful tool for the concentration measurement of light elements in different materials using wavelength dispersive spectrometry [1]. Recently developed multilayers even allow spectroscopy in the Li K range [2]. The high resolution of this spectrometric technique allows observing peak shape alterations and peak shifts depending on the chemical state of the emitting atoms.

Nevertheless, quantitative electron probe microanalysis (EPMA) of light elements is a significant challenge due to the difficulties of signal extraction for low intensities. Therefore, a good choice of measurement parameters and suitable signal treatment, accurate background description, and interference correction are crucial. For light elements, peak to background ratios (P/B) can easily approach unity and special care needs to be paid to the setting of the pulse height analyser (PHA) to avoid noise contribution to the emission peak. Furthermore, the background may have a high curvature and the classical approach of a linear background fit is no longer valid [3].

Our work investigates the EPMA of light elements from Li to F with the aim to determine the best experimentation parameters. The obtained data were used to calculate spectral form factors of emission peaks depending on the material's composition. These form factors help determine the integral measured intensity of the emission and improve quantification accuracy.

Further, the work also focuses on the influence of different background fits on quantification. It proves that a third-degree linear polynomial describes the background better than the commonly used exponential or linear functions. Consequently, the overestimation of P/B values leading to large quantification errors can be avoided.

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