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A new EPMA method for fast trace element analysis in simple matrices

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Abstract

It is well known that trace element sensitivity in electron probe microanalysis (EPMA) is limited by intrinsic random variation in the X-ray continuum background and weak signals at low concentrations. The continuum portion of the background is produced by deceleration of the electron beam by the Coulombic field of the specimen atoms. In addition to the continuum, the background also includes interferences from secondary emission lines, "holes" in the continuum from secondary Bragg diffraction, non-linear curvature of the wavelength-dispersive spectrometer (WDS) continuum and other background artifacts. Typically, the background must be characterized with sufficient precision (along with the peak intensity of the emission line of interest, to obtain the net intensity for subsequent quantification), to attain reasonable accuracy for quantification of the elements of interest. Traditionally we characterize these background intensities by measuring on either side of the emission line and interpolate the intensity underneath the peak to obtain the net intensity. Instead, by applying the mean atomic number (MAN) background calibration curve method proposed in this paper for the background intensity correction, such background measurement artifacts are avoided through identification of outliers within a set of standards. We divide the analytical uncertainty of the MAN background calibration between precision errors and accuracy errors. The precision errors of the MAN background calibration are smaller than direct background measurement, if the mean atomic number of the sample matrix is precisely known. For a simple matrix and a suitable blank standard, a high-precision blank correction can offset the accuracy component of the MAN uncertainty. Use of the blank-corrected-MAN background calibration can further improve our measurement precision for trace elements compared to traditional off-peak measurements because the background determination is not limited by continuum X-ray counting statistics. For trace element mapping of a simple matrix, the background variance due to major element heterogeneity is exceedingly small and high-precision two-dimensional background correction is possible.

Keywords: EPMA, quantitative analysis, microanalysis, trace elements, sensitivity, accuracy, X-ray mapping