American Mineralogist, Volume 106, pages 1717–1735, 2021

Quantitative WDS compositional mapping using the electron microprobe

JOHN J. DONOVAN[1,*], JULIEN M. ALLAZ[2,†], ANETTE VON DER HANDT[3], GARETH G.E. SEWARD[4], OWEN NEILL[5], KARSTEN GOEMANN[6], JULIE CHOUINARD[1], AND PAUL K. CARPENTER[7]

[1]CAMCOR, University of Oregon, Eugene, Oregon, 97403, U.S.A.
[2]Institute of Geochemistry and Petrology, ETH Zürich, 8092 Zürich, Switzerland
[3]Department of Earth Sciences, University of Minnesota, Minneapolis, Minnesota 55455, U.S.A.
[4]Department of Earth Science, University of California Santa Barbara, Santa Barbara, California 93101, U.S.A.
[5]Department of Earth and Environmental Sciences, University of Michigan, Ann Arbor, Michigan 48013, U.S.A.
[6]Central Science Laboratory, University of Tasmania, Hobart, Tasmania 7001, Australia
[7]Department of Earth and Planetary Sciences, Washington University in St. Louis, One Brookings Drive, St. Louis, Missouri 63130, U.S.A.

ABSTRACT

While much progress has been made in electron-probe microanalysis (EPMA) to improve the accuracy of point analysis, the same level of attention has not always been applied to the quantification of wavelength-dispersive spectrometry (WDS) X-ray intensity maps at the individual pixel level. We demonstrate that the same level of rigor applied in traditional point analysis can also be applied to the quantification of pixels in X-ray intensity maps, along with additional acquisition and quantitative processing procedures to further improve accuracy, precision, and mapping throughput. Accordingly, X-ray map quantification should include pixel-level corrections for WDS detector deadtime, corrections for changes in beam current (beam drift), changes in standard intensities (standard drift), high-accuracy removal of background intensities, quantitative matrix corrections, quantitative correction of spectral interferences, and, if required, time-dependent corrections (for beam and/or contamination sensitive materials). The purpose of quantification at the pixel level is to eliminate misinterpretation of intensity artifacts, inherent in raw X-ray intensity signals, that distort the apparent abundance of an element. Major and minor element X-ray signals can contain significant artifacts due to absorption and fluorescence effects. Trace element X-ray signals can contain significant artifacts where phases with different average atomic numbers produce different X-ray continuum (bremsstrahlung) intensities, or where a spectral interference, even an apparently minor one, can produce a false-positive intensity signal. The methods we propose for rigorous pixel quantification require calibration of X-ray intensities on the instrument using standard reference materials, as we already do for point analysis that is then used to quantify multiple X-ray maps, and thus the relative time overhead associated with such pixel-by-pixel quantification is small. Moreover, the absolute time overhead associated with this method is usually less than that required for quantification using manual calibration curve methods while resulting in significantly better accuracy. Applications to geological, synthetic, or engineering materials are numerous as quantitative maps not only show compositional 2D variation of fine-grained or finely zoned structures but also provide very accurate quantitative analysis, with precision approaching that of a single point analysis, when multiple-pixel averaging in compositionally homogeneous domains is utilized.

Keywords: EPMA, WDS, quantitative analysis, X-ray mapping, quantitative mapping