Quantitative phase Analisis: A comparative study of Mo and Cu strictly monochromatic radiations

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A comparison of the Rietveld quantitative phase analyses (RQPA) obtained using Cu-Kα_1, Mo-Kα_1, and synchrotron strictly monochromatic radiations is presented. The main aim is to test a simple hypothesis: *high energy Mo-radiation, combined with high resolution laboratory X-ray powder diffraction optics, could yield more accurate RQPA, for challenging samples, than well-established Cu-radiation procedure(s)*. In order to do so, three set of mixtures with increasing amounts of a given phase (spiking-method) were prepared and the corresponding RQPA results have been evaluated. Firstly, a series of crystalline inorganic phase mixtures with increasing amounts of an analyte was studied in order to determine if Mo-Kα_1 methodology is as robust as the well-established Cu-Kα_1 one. Secondly, a series of crystalline organic phase mixtures with increasing amounts of an organic compound was analyzed. This type of mixture can result in transparency problems in reflection and inhomogeneous loading in narrow capillaries for transmission studies. Finally, a third series with variable amorphous content was studied.

Limit of detection in Cu-patterns, ~0.2 wt%, are slightly lower than those derived from Mo-patterns, ~0.3 wt%, for similar recording times and limit of quantification for a well crystallized inorganic phase using laboratory powder diffraction was established ~0.10 wt%. However, the accuracy was comprised as relative errors were ~100%. Contents higher than 1.0 wt% yielded analyses with relative errors lower than 20%. From the obtained results it is inferred that RQPA from Mo-Kα_1 radiation have slightly better accuracies than those obtained from Cu-Kα_1. This behavior has been established with the calibration graphics obtained through the spiking method and also from Kullback-Leibler distance statistic studies. We explain this outcome, in spite of the lower diffraction power for Mo-radiation (compared to Cu-radiation), due to the larger volume tested with Mo, also because higher energy minimize pattern systematic errors and the microabsorption effect.

Keywords: Limit of quantification, Limit of detection, spiking method, high energy laboratory X-ray powder diffraction