Improvement of Automated Phase Analysis by Scaling on Standard

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Low deviations in diffraction angles are prerequisite for successful application of automated phase analysis on X-ray diffraction data. However, the angular 20 values are often biased by errors in sample position or a finite, but unknown offset in angle for the counter zero position. To overcome these systematic errors without knowing their sources in details we developed a two-step procedure, based on Si-Standard (NIST 640 C) added to the unknown sample. The first step is a Rietveld refinement focusing on the profiles of the Si-lines. As a special feature, Program SIMREF2.8 [1], [2] refines coefficients for a polynomial (3. order) to adjust the observed 20 values to pivots, given by Si lines positions calculated from temperature-dependent Si lattice constants. As output, SIMREF2.8 creates a file with observed intensities, however, with calculated 20 values.

This file is input for the following phase analysis, using program X'PERT Highscore, e.g.. After scaling results become much more lucid and reliable: In MgH_2 +Nb scaling proved to be crucial to obtain correct results for several admixtures and additives.

The authors thank the members of the Laboratorio IDEA, Universitá degli Studi di Trento for the preparation of the MgH_2+Nb samples.

[1] Maichle J.K., Ihringer J., Prandl W., *J. Appl. Cryst.*, 1988, **21**, 22-28. [2] Amann U., <u>http://www.uni-tuebingen.de/uni/pki/simref/simref28(1).exe</u> Keywords: phase analysis, Rietveld refinement, x-ray diffraction