

MEASUREMENT CONDITIONS FOR QUANTITATIVE ANALYSIS USING THE RIETVELD METHOD

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The Rietveld method has been widely used for quantitative analysis of powder samples. However, the influence of the measurement conditions such as the geometry and silt sizes on accuracy and reliability of the analysis results is often overlooked. Those conditions need to be carefully selected to minimize the errors associated with the instrument and the pattern fitting method. This is because the measurement conditions could increase the systematic errors of diffraction angles, decrease diffraction intensities, or cause diffraction profiles that cannot be reproduced and fit well by the commonly used profile functions such as pseudo Voigt and Pearson VII. These changes of the diffraction pattern cause errors of the estimation of integrated intensities and result in inaccurate and unreliable quantitative analysis. In order to see the effects of various measurement conditions, the quantitative analysis results of diffraction patterns collected under different conditions were compared.

A randomly oriented powder sample with less than 30 μm grain size was used to eliminate the errors associated with the sample. The diffraction patterns were collected with different geometry, divergence angle of the incident beam, and resolution of the receiving optics. The Rietveld analysis results of those patterns were compared regarding the accuracy and reliability of the quantity of each phase in the sample. A horizontal sample mount diffractometer, Ultima IV, was used for data collection and powder diffraction pattern analysis software, PDXL, was used for the Rietveld analysis.

The results of comparison and effects of each measurement condition on the quantitative analysis are discussed in this presentation.