

QUANTITATIVE ANALYSIS OF SAMPLES INCLUDING UNKNOWN TRACE PHASES - COMPARISON OF THE RIETVELD AND CALIBRATION CURVE METHODS

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The Rietveld method has been widely used for quantitative analysis of powder samples, mainly because it does not require a pure material of the phase to quantify or special sample preparation to make a calibration curve.

However, the Rietveld method does require complete crystal structure information of all phases included in a sample. If one or more phases remain unknown, their crystal structure information is ignored in the analysis, and this results in a false discrepancy between the observed and simulated patterns and errors in the quantitative analysis. This problem can be avoided by using the calibration curve methods such as the internal standard method that does not require complete crystal structure information. This method uses only two peaks from the phase to quantify and an internal standard material to correct the absorption effects and make a calibration curve. Accurate quantitative values of a selected phase can be obtained even if unknown phases are included in the sample as far as the sample preparation is done carefully.

The comparison between the Rietveld and the internal standard quantitative analysis methods is reported in this presentation. A horizontal sample mount diffractometer, Ultima IV, was used for data collection and powder diffraction pattern analysis software, PDXL, was used for the quantitative analyses.