

A STEPWISE APPROACH FOR THE X-RAY DIFFRACTION DATA IN RIETVELD REFINEMENT

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Consideration of the diffraction data in a way they are collected, i.e., with a step applied by a diffractometer, seems the reasonable way to enhance the structure refinement. With this regard, a number of R-factors to evaluate Rietveld fit are suggested. They account for number of points, automatically referring to an equipment resolution. The new R-factors reflect both goodness of background and peaks fitting while conventional Rietveld R-factors neglect background contribution, sometimes making the R-factors artificially high. The true expressions to the R-factors are:

$$\begin{aligned}
 R_1 &= \sum(|I_{\text{obs}} - I_{\text{calc}}| / I_{\text{obs}}) / N & R_{1w} &= \sum w_i (|I_{\text{obs}} - I_{\text{calc}}| / I_{\text{obs}}) / N \\
 R_2 &= (\sum(|I_{\text{obs}} - I_{\text{calc}}|) / N) / (\sum I_{\text{bragg}} / k_h) & R_{2w} &= \sum (w_i |I_{\text{obs}} - I_{\text{calc}}|) / N / (\sum w_k I_k / k) \\
 R_3 &= (\sum(|I_{\text{obs}} - I_{\text{calc}}|) / N) / I_{\text{bragg}}' & R_{3w} &= \sum (w_i |I_{\text{obs}} - I_{\text{calc}}|) / N / (w_k I_k')
 \end{aligned}$$

where n – number of points;

w_i – weight, $w = 1/\sigma_i$

σ — standard deviance;

I_k – peaks intensities with $I_k \geq 10 \cdot \sigma_k$

k — a number of peaks with intensities $I_k \geq 10 \cdot \sigma_k$

I_k' – a highest peak intensity;

w_k – weight, $w_k = 1/\sigma_k + 1$

The second set of R-factors and goodness of fit are calculated only for $|I_{\text{obs}} - I_{\text{calc}}| - \sigma_i$, i.e.,

$$\begin{aligned}
 R'_1 &= \sum((|I_{\text{obs}} - I_{\text{calc}}| - \sigma) / I_{\text{obs}}) / N & R'_{1w} &= \sum w((|I_{\text{obs}} - I_{\text{calc}}| - \sigma) / I_{\text{obs}}) / N \\
 R'_2 &= (\sum((|I_{\text{obs}} - I_{\text{calc}}| - \sigma)) / N) / (\sum I_k / k_h) & R'_{2w} &= \sum (w(|I_{\text{obs}} - I_{\text{calc}}| - \sigma) / N) / (\sum w I_k / k_h) \\
 R'_3 &= (\sum(|I_{\text{obs}} - I_{\text{calc}}| - \sigma) / N) / I_k' & R'_{3w} &= \sum (w(|I_{\text{obs}} - I_{\text{calc}}| - \sigma)) / N / (I_k')
 \end{aligned}$$

The same way reduced Chi squared is calculated. The third and the fourth sets of R-factors are calculated only for Bragg peaks intensities. The poster demonstrates applications of the suggested R-factors for Rietveld for experimental powder patterns.