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Procedure for Rietveld refinement

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Selection of proper 2D data

Check Debye rings. It should be less spotty and continuous.
Check cake pattern. It should not be wavy.

Data conversion of 2D data to 1D data using Fit2D

Mask spots that have saturated or extremely strong intensity.
Mask 2D area to fan-shape for integration to maximize 2θ range in 1D data.

Correction of diffraction intensity by taking x-ray absorption by diamond into account (using abs_coef_carbon.xls)

Determine I_0 at each data point based on the following equation.

$I_{obs} = I_0 e^{-\mu x}$

μ ; a function of X - ray energy and density of the material (in this case diamond)

x; thickness of the material

Determination of Estimated Standard Deviation (ESD) of intensity at each 2θ data point (using abs_coef_carbon.xls)

Obtain σ at each data point using the equation suggested by Von Dreele (CCP14):

$\sigma = \frac{F\sqrt{I}}{\tan 2\theta}$

σ : ESD

F : constant

Convert 3-columns text data in to text file for GSAS ESD format (using Powder4)

Load data and add phase(s) into GSAS/EXPGUI

Add phase(s) with space group and initial lattice parameters
Add atom(s) and input initial fractional coordinates of the atom(s)
Load the data file of (2θ, I_0, σ) (GSAS ESD format)
Load instrument parameter file (e.g. polarization factor of x-ray = 0.95)

Start Le Bail refinement

Set data limits and excluded regions
Fit background graphically (using typically 10 terms. Background is typically difficult to refine for angle-dispersive data!!)
Refine profile terms (= curve shape parameters in Pseudo-Voigt function)
Refine only GW and LX. Start from GW, then refine both GW and LX.

$\sigma^2 = \mathrm{U}\tan^2\theta + \mathrm{V}\tan\theta + \mathrm{W}$

σ^2 : Variance of the peak in pseudo - Voigt function

(Here U = GU, V = GV, W = GW)

$\gamma = \frac{\mathrm{X}}{\cos\theta} + \mathrm{Y}\tan\theta$

γ : Lorentzian coefficient in pseudo - Voigt function

(Here X = LX, Y = LY)

Refine lattice parameters

Start Rietveld refinement

(1-i) fix lattice parameters and profile terms, refine phase fractions.

(1-ii) fix lattice parameters and profile terms, refine both phase fractions and atomic positions.

(1-iii) fix lattice parameters and profile terms, refine phase fractions, atomic positions, and displacement parameters *U*iso simultaneously (but refinement of displacement parameters tend to yield physically meaningless values, so typically we need to fix *U*iso to reasonable values and never refine).

(2) fix all the above parameters, and refine spherical harmonic terms for preferred orientation correction.

(3) refine all the above parameters together.