

From raw pattern to publication figure — the essentials

A one-page cheat sheet: the formulas, agreement factors, refinement order and figure checklist you actually use. Powder X-ray diffraction (Bragg–Brentano).

Core relations

Bragg's law: $n\lambda = 2d \sin\theta \rightarrow d = \lambda / (2 \sin\theta)$

θ = Bragg angle = $(2\theta)/2$. Cu $K\alpha_1$ $\lambda = 1.5406$ Å.

d → peak position: $2\theta = 2 \arcsin(\lambda / 2d)$

Crystallite size (Scherrer):

$D = K\lambda / (\beta \cos\theta)$, $K \approx 0.9$

β = FWHM in **radians**, instrument-corrected (subtract the width of a standard, e.g. LaB₆/Si). D in the units of λ .
Uncorrected β underestimates size.

Unit cell from d-spacings ($1/d^2$)

System	$1/d^2 =$
Cubic	$(h^2+k^2+l^2) / a^2$
Tetragonal	$(h^2+k^2)/a^2 + l^2/c^2$
Hexagonal	$(4/3)(h^2+hk+k^2)/a^2 + l^2/c^2$
Orthorhombic	$h^2/a^2 + k^2/b^2 + l^2/c^2$

Index ≥ 2 –3 well-resolved peaks, then least-squares the cell.

Rietveld agreement factors

Factor	Meaning	Aim for
R_{wp}	Weighted profile	$< \sim 10\%^*$
R_p	Profile	low
R_{exp}	Expected (best possible)	—
χ^2 / GOF	$(R_{wp}/R_{exp})^2$	~ 1 –2
R_{Bragg}	Structure fit	$< \sim 5$ –8%

*Context-dependent (counts, background). Low R does **not** prove the model is correct — always inspect the difference curve.

Refinement order (stability)

1. Scale factor
2. Background (few terms first)
3. Unit cell
4. Zero shift / specimen displacement
5. Peak shape: $W \rightarrow U, V, W \rightarrow X, Y$
6. Atomic positions
7. Thermal (U_{iso})
8. Occupancies & preferred orientation (last — easy to overfit)

Refine a few parameters at a time; watch the difference plot and R_{wp} after each step.

Publication figure checklist

- ☐ Correct 2θ range; $K\beta$ removed; background handled
- ☐ Axis labels: 2θ (°) & Intensity (a.u.); ticks outside; mirrored axes
- ☐ Label main hkl peaks; state λ / radiation
- ☐ Export **vector (SVG/PDF)** or ≥ 300 dpi PNG
- ☐ Report instrument, radiation, step size
- ☐ Rietveld: show obs (dots), calc (line), difference & Bragg ticks; report R_{wp} , GOF, cell